ANNUAL REPORT OF RESEARCH AND TECHNOLOGIC WORK ON COAL, FISCAL YEAR 1949

By Arno C. Fieldner and Sidney Gottlieb

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by

Arno C. Fieldner[1] and Sidney Gottlieb[2]

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INTRODUCTION

The research and technologic work of the Bureau of Mines on coal and coal products in the period July 1, 1948, to July 1, 1949, is summarized in this report. Based largely upon publications issued during the fiscal year, it constitutes the fourteenth in a series of such resumes. Further details of the research conducted may be found in the publications given in the footnote references, though in some cases research data not published elsewhere are presented herein.

For the first time, results of research in the experimental coal mine and on explosives and gas and dust explosions are omitted from this report. These data will be included in Bureau of Mines Report of Investigations 4667, entitled, "Annual Report of Research and Technologic Work on Explosives, Explosions, and Flames, Fiscal Year 1949."

As petroleum consumption continued to trend upward, strong interest in the synthetic liquid fuels program of the Bureau of Mines continued to be manifested. This program and the salient features of a projected synthetic liquid fuels industry, based principally upon coals as a raw material, were discussed on several occasions.3/, 4/, 5/ A complete report on Bureau of Mines research in the calendar year 1948 on developing processes for production of synthetic oil from coal was rendered to the 81st Congress.6/ A discussion7/ of our reserves of solid fuels covered our present reserve position and the potentialities of those reserves.

Construction of the Anthracite Research Laboratory,8/ authorized by the 80th Congress, was well under way by the end of the fiscal year at Schuylkill Haven, Pa., on a site of approximately 16 acres donated by the Philadelphia & Reading Coal & Iron Co. and Schuylkill County. Occupancy was expected in January 1950. Designs for a laboratory building for lignite research, to be erected on a site donated by the University of North Dakota on its campus at Grand Forks, were completed (see fig. 1).

Summary

With coal supplies exceeding demands, the value of Bureau of Mines research on the mining, preparation, and utilization of coal became increasingly apparent. Increased efficiency and improved quality became the keynotes of the research program, along with conservation and safety.

The Bureau continued to provide service to other Federal agencies on fuels-utilization problems in its role of consultant on the purchase of fuel and fuel-burning equipment, tests of fuels and boiler-room equipment, and boiler-water chemical treatment. In providing these services, 264 boiler-water test kits, 12,005

3/ Doherty, J. D., Coal for Synthetic Liquid Fuels: Min. Eng., vol. 1, No. 4, 1949, pp. 116-124.
4/ Cattell, R. A., and Doherty, J. D., Synthetic Liquid Fuels: (in part) Producers Monthly, vol. 12, 1948, pp. 21-29.
5/ Fieldner, A. C., Frontiers of Fuel Technology: Fuel, vol. 28, 1949, pp. 19-21; Chem. and Eng. News, vol. 26, 1948, pp. 1700-1701.
6/ Synthetic Liquid Fuels 1948 Annual Report of the Secretary of the Interior, Part I. - Oil from Coal: Bureau of Mines Rept. of Investigation 4456, 1949, 62 pp.
7/ Fieldner, A. C., Solid Fuels.....and Their Suitability for Production of Liquid and Gaseous Fuels: Oil and Gas Jour., vol. 47, 1949, pp. 138-140, 148, and 145.
8/ Buch, J. W., New Anthracite Research Laboratory at Schuylkill Haven, Pa.: Reading Railroad Mag., vol. 12, 1948, pp. 3-5, 24.

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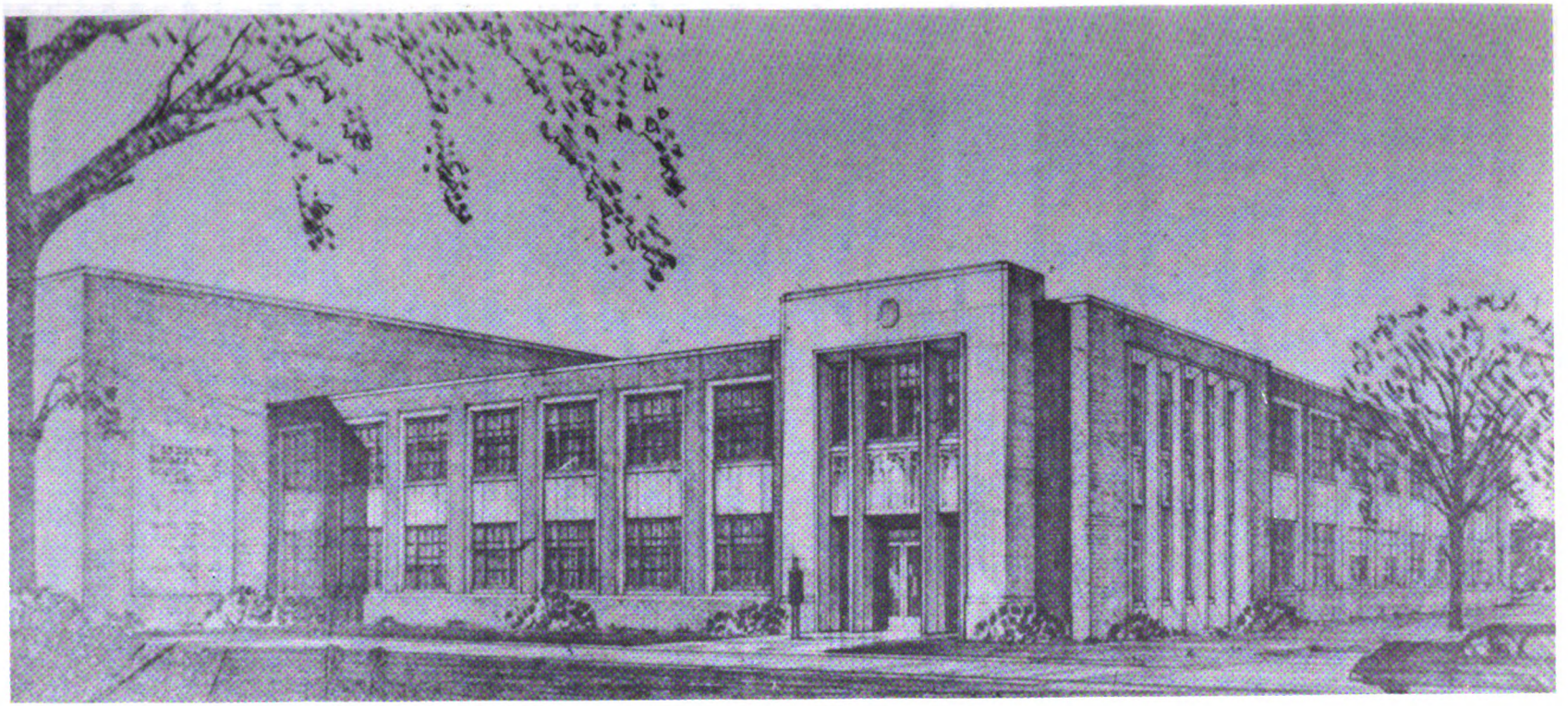

Figure 1. - Lignite research laboratory, Grand Forks, N. Dak.

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bottles of chemical reagents, and 10,040 test kit replacement items were distributed to Federal boiler installations; 742 samples of boiler water and 7,057 samples of coal from Government purchases, tipple inspections, and export shipments were analyzed. As in previous years, fuel-engineering services were most productive, yielding savings in operation costs at numerous Government fuel-burning and boiler installations. Boiler-water research demonstrated the value of certain chemicals in controlling corrosion in steam lines. Control of causticity was proved effective in preventing boiler-seam cracking.

Many data were added to our knowledge of the fundamental properties and constitution of coal by petrographic studies of coals from Maryland, Colorado, North Carolina, Alabama, California, and Wyoming.

To meet the needs of fuel-short and strategic areas, core-drilling investigations of Alaskan coal deposits were continued. The Eska Creek area was found to be unsuitable for the development of a mechanized mine, and diamond drilling in the Wishbone Hill area southwest of Jonesville, Alaska, was begun. Reconnaissance of other coal fields yielded data on which future investigations will be based.

Diamond-drilling investigation of the Coosa coal field, Alabama, revealed a reserve of coal in the Fairview bed estimated at 14,580,000 tons, of which 11,660,000 tons should be recoverable. In the Minnesota Creek area, Gunnison County, Colo., minable coal reserves totaling 781,000,000 tons were found, of which 624,800,000 tons may be assumed to be recoverable. Other drilling projects were completed in the Castleman Basin, Maryland, in the Deep River coal field, North Carolina, and in the Coal Creek district of Colorado.

Confronted by depletion of our coking-coal reserves and the necessity for accurate information about remaining reserves to serve as a basis for national security planning, the Bureau of Mines undertook a field investigation of known minable coking-coal reserves. During the year, field parties collected information in Pennsylvania, West Virginia, and Kentucky. Under the State Department program of cooperation with other American republics, the Bureau of Mines studied and reported on the coal fields and industry of Brazil and Chile. To increase efficiency in coal mining, the Bureau studied the extraction of pillars with mechanized equipment as well as overall mining practice in Arkansas and the Missouri River Basin.

The suitability of German shearing machines in increasing recovery of anthracite was demonstrated in underground operations, and tests of a German pneumatic packing machine were successful.

Bureau of Mines research on the use of Diesel engines underground was summarized and indicated that adequate ventilation and the provision of flame protective devices are essential to safe operation. Schedules for Bureau of Mines testing of Diesel mine locomotives for permissibility were issued. The Bureau of Mines continued to test mine atmospheres for toxic constituents, a program that involved the analysis of 15,000 samples of mine air and the examination of 750 samples of air-borne dusts. After testing respiratory protective devices, 6 new approvals and 37 extensions of approval were granted.

In its investigation of the prevention of flooding of anthracite mines, the Bureau completed a survey of underground water pools and assembled information from thousands of boreholes in the anthracite fields. Possible sites for drainage tunnels were considered. Testing of electrical equipment for permissibility for use in coal mines was continued, and 53 approvals were granted. A notable contribution to safety

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in coal mines was made during the year when the Bureau demonstrated the practical application of roof support in coal mines by means of suspension roof supports.

The need for improvement in coal-preparation practices continued to be felt as coal mining became more and more mechanized. To meet this need, a Bureau-developed process for cleaning and recovering fine coal was installed commercially at two mines in Alabama, and a cyclone-thickener unit was installed at a preparation plant in West Virginia. The usefulness of both installations was demonstrated during the year. The status of coal-washing practices in Washington, Oregon, and Alaska, where coal-preparation problems are particularly acute, was summarized, and a study of sampling coal for float-and-sink tests promised to facilitate future research. Paralleling the study of known minable reserves of coking coal, research was conducted on the preparation characteristics of high-sulfur coking coals in southwestern Pennsylvania and northern West Virginia. Methods of producing extremely clean coal suitable for electrode carbon in Germany were reviewed, and a bibliography of publications on the briquetting of coal was prepared.

With increasing industrialization in the west, emphasis has been placed on the development of satisfactory methods of efficiently utilizing the huge reserves of low-rank coals in that area. Pilot-plant tests of drying processes for low-rank coals were particularly promising. Millions of tons of lignite now being excavated from a dam site in North Dakota were stored satisfactorily by Bureau-developed methods.

To promote conservation in the domestic use of coal, a research program to assist householders in selecting coal-burning equipment and to assist coal producers in preparing coal for domestic stoker use was completed. Conservation in the utilization of fuel was furthered also by research on the reactivity of solid fuels and a study of furnace performance.

The reserves study and preparation phases of the Bureau's program to increase our available sources of coking coals were supplemented by continuation of the survey of the carbonizing properties of American coals, in which particular emphasis was placed upon coals and blends of coals suitable for the manufacture of metallurgical coke. Various properties of 37 coals and 28 blends were determined during the year, including the coke-, gas-, and byproduct-making properties of 20 coals. The blending of Appalachian coals was studied, and the carbonizing properties of Chilean coals were determined.

Research was continued on the complete gasification of coal by the Lurgi process, and further progress was made in developing a satisfactory externally heated, continuous, annular retort for the gasification of lignite. Continuing its investigation of the feasibility of utilizing coal without mining by means of underground gasification, the Bureau of Mines undertook its second field-scale project at Gorgas, Ala., in cooperation with the Alabama Power Co. The installation was planned in five straight-line sections, the first of which, 300 feet long, was ignited by means of a thermite hand grenade in March 1949. The project has been operated continuously since that time.

Development of methods for the production of liquid fuels from coal continued to be a major part of the Bureau of Mines research program. Laboratory-scale experimentation on the production of synthesis gas demonstrated that all grades of coal can be continuously gasified successfully to furnish synthesis gas from which liquid fuels can be synthesized. In the course of this work, a pneumatic feeder was developed which provides a method of feeding solids to a reaction zone at a uniform rate. The other most promising process for the production of liquid fuel from coal, hydrogenation of coal, was the subject of laboratory and pilot-plant study, and the results

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were most encouraging. Research on the liquid-phase and vapor-phase hydrogenation of coal, the testing of catalysts, and the separation and identification of coal-hydrogenation products were important aspects of the work. Other types of Fischer-Tropsch processes have been developed for the liquefaction of coal by the hydrogenation of carbon monoxide. Much attention has been paid to the role of various catalysts in these processes.

Based upon the laboratory and pilot-plant research on the gas synthesis and coal-hydrogenation processes, two coal-to-oil demonstration plants were dedicated at Louisiana, Mo., in May 1949. Although these plants were not completed to the full operation stage, successful unit operations have been carried out in both.

Acknowledgments

This report includes work done under the technical direction of the following members of the staff of the Bureau of Mines:

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University of West Virginia

ORIGIN, COMPOSITION, AND PROPERTIES OF COAL

Inspection, Sampling, and Analysis

At the time contracts for coal were let for the fiscal year 1949, demand was in excess of the supply, and Government agencies were not always able to obtain coal of the desired quality. Coal was purchased on a guaranteed-analysis basis, including penalties for delivery of substandard coal, by the Bureau of Prisons, Department of Justice, Bureau of Indian Affairs, Department of the Interior, and for export. Other departments purchased coal at a fixed price subject to adjustment by negotiation for delivery of substandard coal, or at a fixed price not subject to adjustment for quality. Contracting for coal for the fiscal year 1950 began in March, and by that time the market had changed from a "seller's" to a "buyer's" market, resulting in a flood of bids on Government requirements. All departments issued specifications calling for bids on a guaranteed-analysis basis, including penalties for delivery of substandard coal. In general, each department purchases its own coal supply, except that the Navy Department purchases coal for the Army and the Bureau of Federal Supply purchases a supply for other agencies whose requirements are small.

Coal requirements of Federal agencies for the fiscal year were estimated at approximately 5,500,000 tons. Owing to the large number of bids for 1950 requirements, there was a large increase in requests from Government agencies for analyses for use in evaluating coal bids. In addition, recommendations were made for awards of contracts for a number of agencies.

For the use of various Government agencies in making coal purchases and for public information, 756 tipple samples were collected at 191 mines in four States.

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At the close of the year, requests for sampling more than 600 mines were on file. For research on carbonization, free swelling, grindability, gasification, and other properties of coal, 183 samples were collected in 158 mines. Approximately 500,000 tons of coal purchased by the War Department on a guaranteed-analysis basis for shipment to Japan were inspected and sampled at Philadelphia, Pa.; Baltimore, Md.; and Norfolk, Va.

Upon request of the Air Forces, schools for coal samplers, attended by samplers from airfields in surrounding territories, were conducted at Griffiss Field, Rome, N. Y.; Wright-Patterson Field, Dayton, Ohio; Warner Robins Field, Warner Robins, Ga.; and Hill Field, Ogden, Utah.

Proximate or ultimate analyses were made of 7,057 samples of coal and coke from Government purchases, tipple and breaker inspections, and coal exported to Europe and Japan. The greater portion of these samples came from branches of the Department of National Defense; the rest were from civilian agencies such as post offices, Federal buildings, Indian Affairs, Federal Bureau of Supply, Veterans Administration, and others.

Research and test work of the Bureau required analysis of 8,428 samples of coal or products derived from coal in connection with investigations on the mining, preparation, and utilization of coal. In cooperation with the Economic Cooperation Administration, coals from Greece and Korea were analyzed.

The inspectors of the Coal Mine Inspection Branch of the Health and Safety Division submitted 8,631 samples of road, roof, rib, and gob dusts from the larger shipping coal mines. These samples represent 1,693 inspections and approximate the number of mines in which dusts were taken for analysis. The samples are usually received, analyzed, and the report sent out the same day to the inspectors for their use in determining the safety of operations in the mines and in formulating recommendations where changes are indicated.

The total of 24,116 samples of coal, coke, char, tar, coal-mine dusts, and related materials that were analyzed represents the largest number of samples ever handled by the laboratory, exceeding the previous high year of 1943 by 6.4 percent and last year by 19.3 percent. In addition, investigation of the deterioration of the free-swelling index of coal in storage was continued.

Analyses of Miscellaneous Materials

Chemical analyses of 110 samples of miscellaneous materials were made in connection with various investigations of coal utilization and safety in mines.

This work included complete analyses of 38 samples of coal ash from coals being investigated for use in the synthetic liquid fuels program and four samples of ash from Kentucky coal burned in a boiler furnace during heat absorption tests. Analyses of ash from seven samples of roof and floor rock adjoining the coal bed in Underground Gasification Project No. 2, Gorgas, Ala., showed that the ignited rock had a high silica content. Complete analyses of 24 boiler scales or water-formed deposits and of 10 boiler compounds collected from steam plants of various Government agencies were made to assist in the preparation of recommendations for improved feed-water treatment at these plants.

Examination of eight samples of rock dusting materials showed that one sample, a limestone from Kentucky, would be suitable for rock-dusting coal mines. The other materials tested did not meet all the requirements for a satisfactory rock dust. In

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connection with health and safety investigations, an analysis was made of blast-furnace slag proposed as a substitute for sand as traction material for mine locomotives. Other analyses showed a high silica content in two samples of rock collected during an inspection of dust hazards in a quarry producing aggregate for concrete.

Sixteen other materials were analyzed or tested in connection with investigations of carbonization, complete gasification, and combustion of coal.

Constitution, Properties, and Analytical Methods

Petrographic Studies of Coal

Petrographic examinations were made of 168 cores, totaling 589 feet, recovered from coal investigations in Garrett County, Md.; Eska, Alaska; Gunnison and Routt Counties, Colo.; Chatham County, N. C.; Walker County, Ala.; Contra Costa County, Calif.; and Johnson County, Wyo. Laboratory study of the cores involved complete megascopic examination and preparation of descriptive log strips used in correlating the drilling data and the various coal beds encountered in the test drilling. On the basis of petrographic interpretation of the cores, 390 feet of coal was selectively sampled for special chemical and physical tests.

Complete thin-section analyses were made of the following Colorado coals: 70-inch column sample from the Lennox bed, Edna mine, Routt County; 124-inch column sample from the No. 4 bed, King mine, near Paonia, Delta County; and a drill-core sample representing a 12-foot, 11-inch coal bed from the Coal Creek area, Gunnison County. Their petrographic composition is given in table 1. The results show that these coals consist principally of anthraxylon and translucent attritus. Their low content of opaque attritus and fusain, which are components classed as relatively inert to chemical reactions, indicate that the coals would be suitable for preparation of synthetic liquid fuels by the hydrogenation process.

TABLE 1. - Petrographic composition of Colorado coals

Petrographic components	Edna mine	King mine	Drill core
Anthraxylon	49.9	48.3	45.6
Translucent attritus	45.4	46.0	47.4
Opaque attritus	2.1	2.1	4.0
Fusain	1.4	1.9	3.0
Translucent mineral matter ...	1/.1	1/1.7	-
Pyritic mineral matter	1.1	2/0.1	-

1/ Principally calcite and clay minerals.
2/ Less than.

Cores from the drilling investigations in the Minnesota Creek area, Gunnison County, Colo.,9/ were studied megascopically, and cores from two test holes were selected as being representative of the coal beds cored in the various test holes and were analyzed microscopically by the thin-section method. The results showed the coal in all eight beds to be more than 3 feet thick in one hole and the seven

9/ Toenges, Albert L., Dowd, James D., Turnbull, Louis A., Davis, J. D., Smith, H. L., and Johnson, Vard H., Reserves, Petrographic and Chemical Characteristics, and Carbonizing Properties of Coal Occurring South of Dry Fork of Minnesota Creek, Gunnison County, near Paonia, Colo., and the Geology of the Area: Bureau of Mines Tech. Paper 721, 1949, 48 pp.

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beds in the other hole to be remarkably uniform in petrographic composition. On the basis of thin-section analyses, these coals were classified as bright, with relatively low amounts of opaque attritus and fusain and only a very small amount of mineral matter, usually in the form of dispersed clay minerals, fine quartz grains, and pyrite. Carbonization tests showed that the coals examined produce a rather inferior grade of coke, which is not equal to that made from the Lower Sunnyside coal in Utah.

Cores recovered in diamond-drilling investigation of the Fairview and Coal City basins in St. Clair County, Ala., were studied in the laboratory.10/ A column sample and drill-core sample from the Fairview bed were thin-sectioned and analyzed microscopically. On the basis of the microscopic analyses, the Fairview bed is classified as bright coal. The anthraxylon content ranges from 57 to 64 percent and is associated with 19 to 20 percent translucent attritus. Opaque attritus is not particularly high, ranging from 8 to 9 percent, but an unusual feature of the petrographic composition is the relatively large amount of fusain present - 8 to 15 percent. Coal beds normally rarely exceed 3 percent fusain content. The microscopic study showed that most of the mineral matter, which reached rather high concentrations in some layers, consists of pyrite, calcite, and clay minerals. The pyrite and calcite, principally of secondary origin, occurred in lenses and veinlets, many of which are visible to the naked eye. The clay minerals were present as dispersed detrital particles in the attrital layers of coal. Evidence that the Fairview bed had been subjected to the stresses of crustal movement was seen in the arching of certain anthraxylon layers and minute faulting and fracturing.

Petrographic analyses were made of float-and-sink fractions of three samples of broken coal from the Pittsburgh bed to determine whether coal-preparation methods, particularly gravity separation, could be used for concentrating in the low specific-gravity fractions certain coal components such as anthraxylon and bright attritus considered superior for hydrogenation. Also, it was desired to know whether a maximum rejection could be achieved in the heavy specific-gravity fractions of coal less favorable for hydrogenation, such as dull attritus with high content of opaque matter, fusain, and associated free mineral matter. The three sized fractions (1-1/2-inch by 3/8-inch, 3/8-inch by 14-mesh, and 14-mesh by 100-mesh), prepared by crushing and screening the mine samples, were classified in gravity separation liquids of 1.3 and 1.6 specific gravities. The three float-and-sink fractions were crushed to pass 8-mesh and, with the untreated minus 100-mesh fraction, were screened on 10 sieves, from 10-mesh through 200-mesh. The composition of each screen fraction was determined by petrographic method of particle identification and count using an ore-dressing microscope.

The data show that anthraxylon and bright attritus are concentrated in coal that floats on separation liquids of 1.3 to 1.6 specific gravities. Fusain is concentrated in minus 100-mesh fines and mineral matter in the 1.60 sp. gr. sink. The results indicate that coal can be improved for hydrogenation by preparation methods.

10/ Toenges, Albert L., Turnbull, Louis A., Jolley, Theodore R., Shields, Joseph J., Smith, H. L., O'Donnell, H. J., Cooper, H. M., Abernethy, R. F., Gandrud, B. W., Riley, H. L., and Rothrock, Howard E., Investigation of Coal Deposits in the Fairview and Coal City Basins of Coosa Field, St. Clair County, Ala.: Reserves, Petrography, and Chemical Properties of Coals; Washability Characteristics of Coal from Fairview Bed; and Geology of the Area: Bureau of Mines Tech. Paper 719, 1949, 104 pp.

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A complete thin-section analysis was made of an 81-inch column of lignite obtained from the American Lignite Products Co.'s mine in Amador County, Calif. The column sample represents the upper part of a lignite bed which ranges in thickness from 1 to 12 feet. The sample is of particular interest because the deposit is being exploited as a source of raw material for a wax-extraction plant, which is the only one of its kind operating in the United States. Petrographic analysis of this lignite permitted comparison with similar lignites from deposits occurring in southern Arkansas and at one time used to a limited extent for wax extraction in a lignite-processing plant now abandoned. The Amador County lignite proved to be an attrital type containing an extremely high proportion of translucent attritus. Approximately one-quarter of the translucent attritus consisted of yellow translucent matter of relatively small particle size, predominantly minus 0.050 mm. The general petrographic composition differed from the Arkansas attrital lignites in much lower content of resin bodies and considerably finer particle size of the attritus ground mass.

Examination of coal that has been reduced by various stages of preparation into granular form is the subject of continuing investigation in the coal petrography laboratory. Despite considerable work done, a standard method of analysis has not been developed, as the problems involved are not simple. Various types of plastics and waxes, such as bakelite and carnauba wax, are used with heat and pressure in a small hydraulic laboratory press to bind granular coal into so-called briquettes about 1 inch in diameter and up to 3 inches in length, which are used for preparing thin sections. The crushing effect of the press on particles of coal was studied, and it was found that with the use of certain high-melting waxes, such as carnauba, extremely high pressures not only are not necessary but are detrimental in that a high percentage of particles are crushed to very fine sizes, making microscopic work very difficult. Tests of the crushing effect of the hydraulic press were made on samples of anthraxylon and splint coal closely sized into 14- by 20-mesh and 65- by 100-mesh fractions, with pressures of 500, 1,000, 2,000, and 3,000 p.s.i. The least measurable effect of crushing was noted in briquettes made with the 65- by 100-mesh fractions using pressure of 500 p.s.i. Anthraxylon was more responsive to the crushing action than splint coal. It was concluded that for microscopic examination using magnifications from 60 to 150 power, satisfactory thin sections can be prepared from briquettes made with granular coal sized 65- by 100-mesh and formed at pressure of 500 p.s.i.

Determination of Chlorine in Coal

A method of determining chlorine in coal, as published in British Standards Institution No. 1016, was investigated.

The British method is a modified Eschka procedure whereby a 5-gram sample of coal is ignited with sodium carbonate at 475° C. for 20 to 24 hours. The sample is then acidified with nitric acid and chlorine determined by the Volhard method. Chlorine was determined in three coals by this method and, for comparison, by a water-extraction method and an oxygen-bomb method in which chlorine is determined in the bomb washings. Results are given in table 2.

The percentages of chlorine are recorded to three decimal places to show variations more clearly. Previous work has shown that for some coals, water extraction does not recover all of the chlorine, but for these coals virtually all was recovered. Results by the British method agree with those obtained by the bomb method for two of the samples and are slightly higher for the third. It was concluded that the British procedure is a practical method of determining chlorine in coal.

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TABLE 2. - Chlorine content of coal

Source of coal			Percent chlorine in coal		
State	County	Bed	British method	Oxygen-bomb method	Water-extraction method
Ill.	Perry	No. 6	0.019		
			.014	0.021	0.010
			.016	.013	.013
		Ave.	.016	.017	.012
Ky.	Muhlenburg	No. 9	0.024		
			.027	0.026	0.019
			.024	.024	.020
		Ave.	.025	.025	.020
W. Va.	Marshall	Pittsburgh	0.224	0.182	
			.227	.187	0.192
			.223	.207	.195
		Ave.	.225	.192	.194

Free-Swelling Index of Coal

In the Standard Method of Test for Free-Swelling Index of Coal (A.S.T.M. Designation: D 720 - 46), the specifications provide that the temperature of the inner surface of the bottom of the crucible used in carbonizing the coal shall reach 800 ± 10° C. in 1-1/2 minutes and 820 ± 5° C. in 2-1/2 minutes after placing the crucible over the gas flame. The crucible used shall also meet certain dimensional requirements, the weight of the crucible being within the limits of 11.0 to 12.75 grams. At the normal gas pressure in some laboratories, the required crucible temperatures are difficult to attain. Also, it is often not easy to purchase silica crucibles that will meet the weight specifications. Because of these difficulties, tests were made as to the effect of varying the carbonizing temperature and crucible weights to determine whether tolerances in the specifications could be increased.

Free-swelling tests of three samples of coal were made at 750° C., 775° C., and 800° C. Table 3 shows the results of the tests made at these temperatures. In general, the carbonizing temperature has a significant effect upon free-swelling results, the swelling indexes increasing at higher temperatures. Therefore, the specified temperatures of the standard method must be maintained.

Tests of the effect of crucible weight showed that within the range of weight limits studied (10.7 to 15.3 grams), free-swelling results were not significantly affected. It was concluded that if the crucibles used permitted the proper temperatures to be reached with no change in burner adjustment during tests with crucibles of various weights, the weight limits specified in the standard method probably could be increased.

TABLE 3. - Effect of temperature upon free-swelling indexes of coal

Source of coal examined			Free-swelling index at crucible temperature, °C.		
State	County	Bed	750	775	800
Illinois	Madison	No. 6	3	3	3
Pennsylvania	Somerset	E	6-1/2	7	8
West Virginia	Logan	Chilton	7	8	8-1/2

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Determination of Petrographic Components in Coal by Thin-Section Examination

An evaluation of the Bureau of Mines method of determining the petrographic components of coal by examination of thin sections showed that a high degree of accuracy is attained in the microscopic determination of coal components.11/ Reproducible results can be obtained irrespective of make of microscope or power of magnification used and the technician performing the work, provided the standard technique is followed with reasonable exactness. The usefulness of petrographic analyses in evaluating coal for certain purposes is discussed. It is pointed out, for instance, that quantitative determinations of the components in coal enable predictions to be made regarding amenability of the coal to hydrogenation and expected yield of liquid products. Exactness in predictions is related to completeness and accuracy of the petrographic analysis.

COAL MINING

Coal Investigation

Alaska

Coal deposits in south-central Alaska and the Kenai Peninsula were investigated by engineers of Bituminous Coal Mining Section.12/

The investigation covered reconnaissance to serve as a basis for plans for detailed investigations of minable coal reserves (see fig. 2). The Matanuska Valley Coal Field is in the valleys of the Matanuska River and its tributaries and their separating ridges. Coal exposures occur in an area approximately 25 miles long by 7 miles wide and parallel the general trend of the Matanuska Valley.

The Lower Matanuska Valley bituminous-coal field occurs in the prominent Wishbone Hill syncline, which extends 7 to 10 miles in a southwestern direction from Eska to Moose Creek (see fig. 3). Bituminous coal is restricted to the Chickaloon formation of Tertiary age; and the coal beds generally occur in the upper 1,400 feet of the formation. At present, the Evan-Jones mine, at Jonesville, on the Matanuska branch line of the Alaska Railroad, about 17 miles east of Palmer by highway and 58 miles from Anchorage via Palmer is the only mine in operation in the Matanuska coal field. The bituminous coal from this mine ships and stores well and is preferred to the subbituminous coal of the Nenan field to the north.

The Moose Creek district lies on the north limb of the Wishbone Hill syncline in the western part of the Matanuska field, about 5 miles west of Jonesville. The Pioneer, Baxter, Rawson, Alaska-Matanuska, and Premier mines have been abandoned owing to physical conditions in and surrounding the beds.

The Eska Creek area is adjacent to the abandoned Eska mine and east of Eska Creek. In an investigation of this area by the Bureau of Mines by diamond drilling, 9 holes totaling 5,158 feet were completed during the year. Data from the completed investigation indicate that the area is not favorable for the development of a mechanized mine.

11/ Parks, B. C., and O'Donnell, H. J., Determination of Petrographic Components of Coal by Examination of Thin Sections: A.I.M.E. Tech. Pub. 2492, Coal Technology, Nov. 1948, 17 pp.

12/ Toenges, Albert L., and Jolley, Theodore R., Investigation of Coal Deposits in South Central Alaska and the Kenai Peninsula: Bureau of Mines Rept. of Investigations 4520, 1949, 37 pp.

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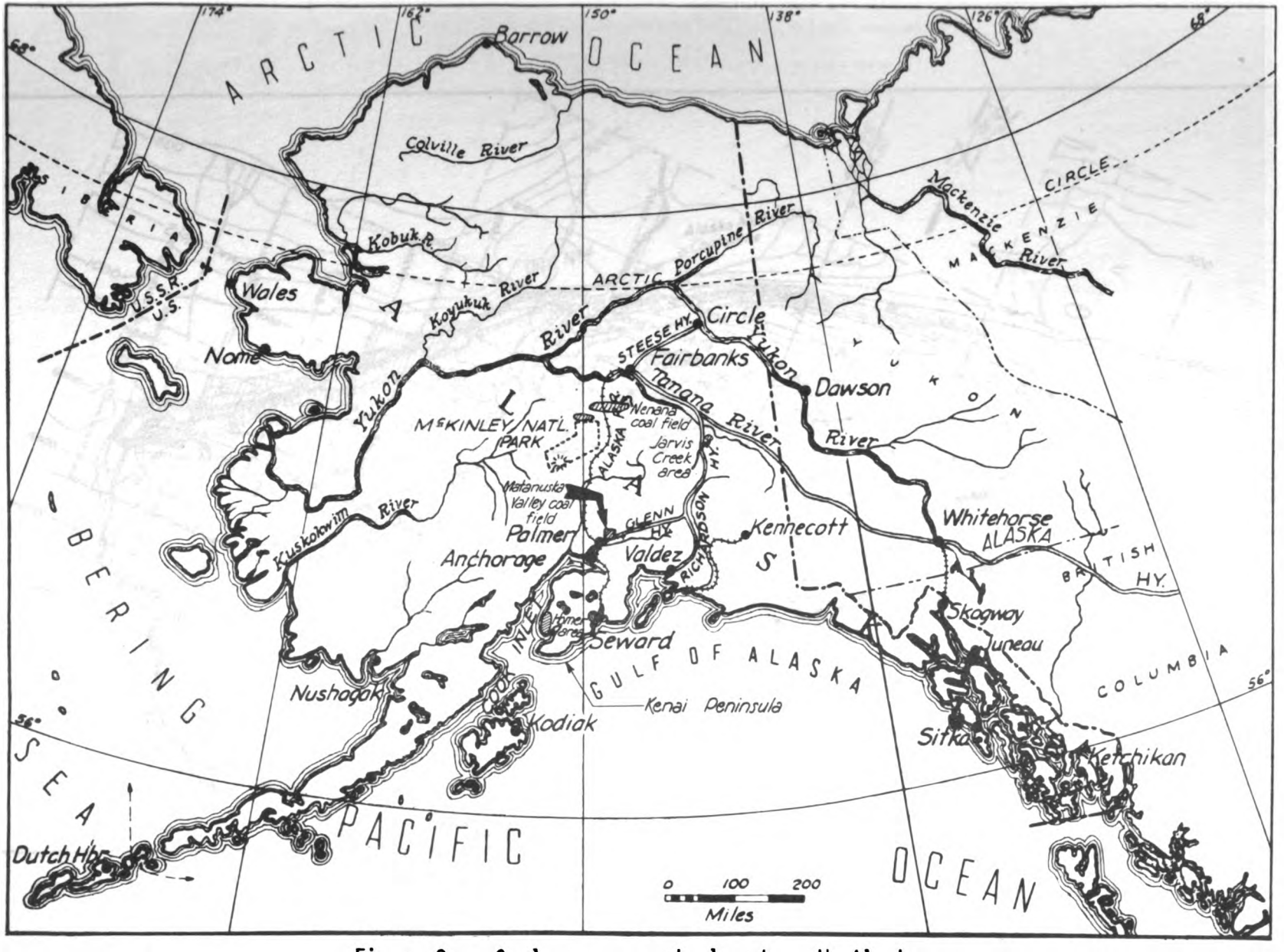

Figure 2. - Coal areas, central and south Alaska.

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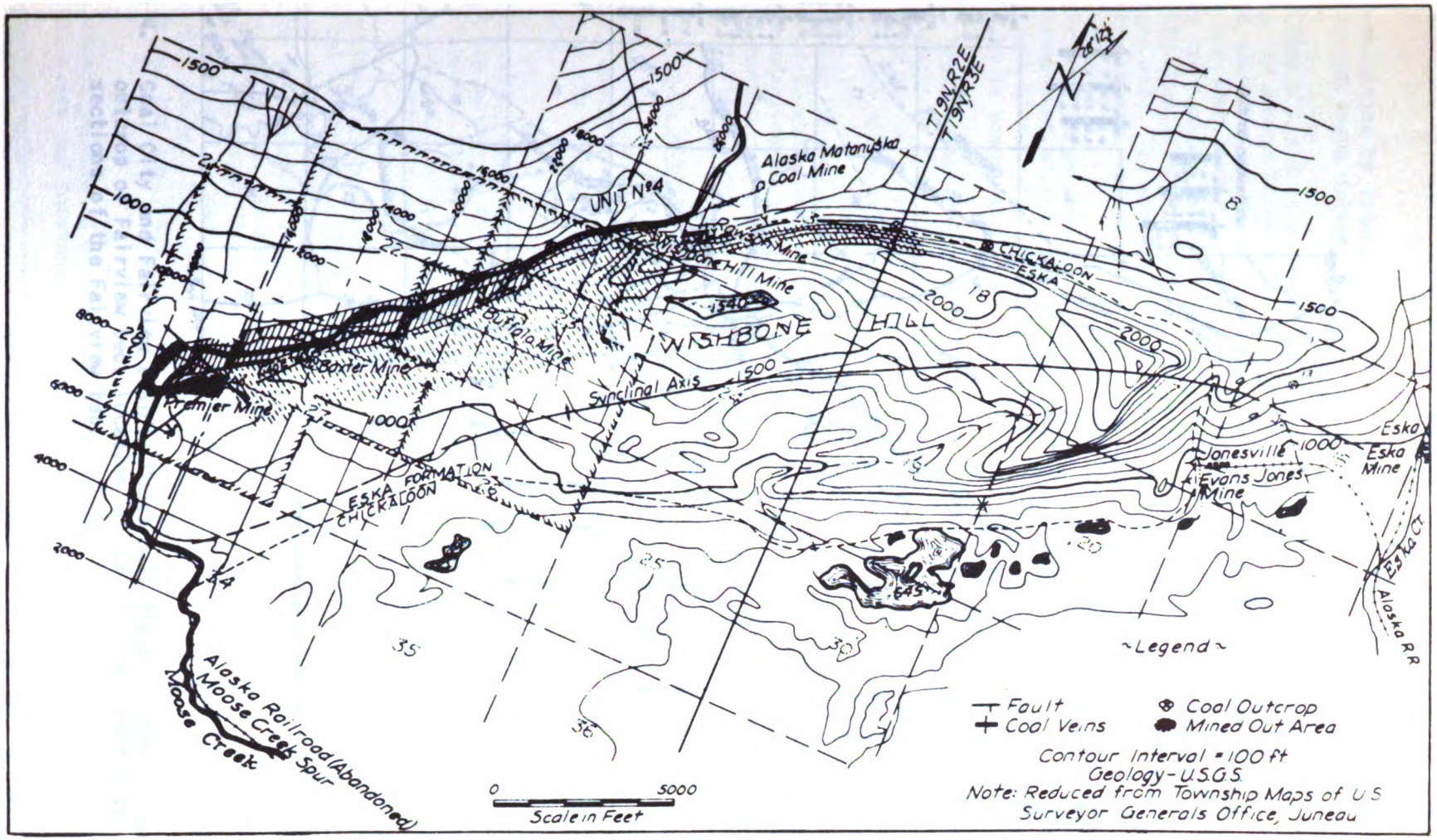

Figure 3. - Moose Creek coal district, Alaska.

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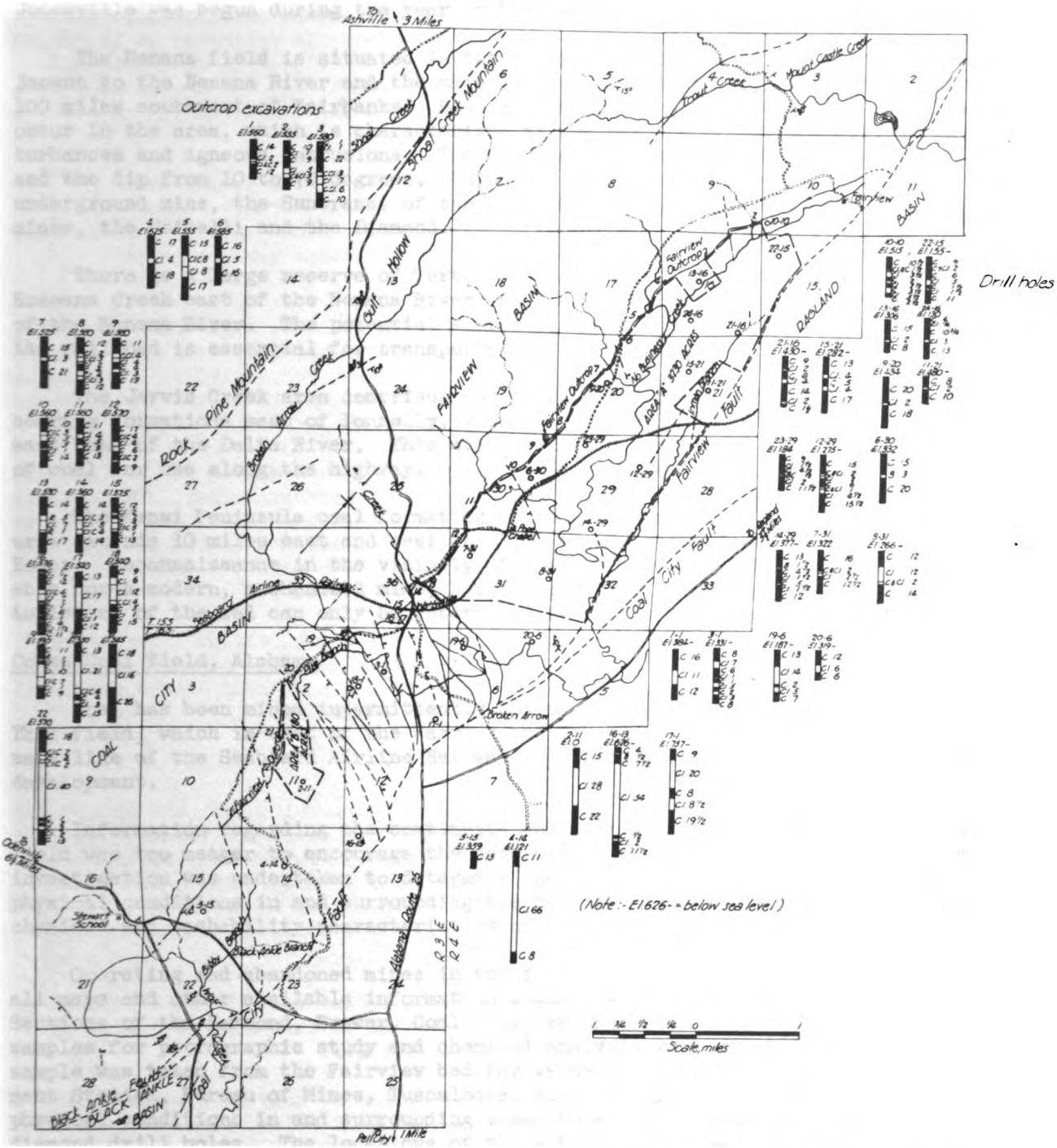

Figure 4. - Coal City and Fairview basins, Coosa coal field, Alabama, showing outcrop of Fairview bed, location of drill holes, test pits, and sections of the Fairview bed.

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Investigation by diamond drilling of an area in Wishbone Hill southwest of Jonesville was begun during the year by the Bureau.

The Nenana field is situated in the northern foothills of the Alaska Range adjacent to the Nenana River and the main line of the Alaska Railroad and approximately 100 miles southwest of Fairbanks. Beds of low-rank subbituminous coal and lignite occur in the area, which is characterized by the absence of extreme structural disturbances and igneous intrusions. The thickness of the beds ranges from 6 to 50 feet and the dip from 10 to 70 degrees. In this field, coal has been produced from one underground mine, the Suntrana, of the Healy River Coal Corp., and from two strip mines, the Usibelli and the Diamond.

There is a large reserve of Tertiary coal-bearing formation along Lignite or Hoseana Creek east of the Nenana River and Alaska Railroad, which is on the west bank of the Nenana River. The potential source of coal is amenable to development, and the railroad is essential for transportation.

The Jarvis Creek area comprises about 12 to 15 square miles of Tertiary coal-bearing formations east of Donnelly, which is on the Richardson highway and on the east bank of the Delta River. This area should be considered as a potential source of coal for use along the highway.

The Kenai Peninsula coal formation contains several coal beds, and the probable area extends 10 miles east and west and 3 miles north and south in the vicinity of Homer. Reconnaissance in the vicinity of Homer indicated physical conditions favorable for a modern, mechanized mine, but the extent of minable reserves and characteristics of the bed can only be determined by an investigation by diamond drilling.

Coosa Coal Field, Alabama

Coal has been mined intermittently for many years in the Coosa field of Alabama. This field, which is east of the Warrior and Cahaba coal fields, is traversed by the main line of the Seaboard Airline Railway and is adjacent to the proposed Coosa River development.

Information regarding the continuity and character of the coal beds in the Coosa Field was too meager to encourage the development of modern mechanized mines, and an investigation was undertaken to determine the thickness and extent of the coal beds, physical conditions in and surrounding the beds that would influence mining, and the chemical and washability characteristics of the coals.13/

Operating and abandoned mines in the field, where accessible, were examined, and all maps and other available information regarding abandoned mines were obtained. Sections of the Hammond, Brewer, Coal City, and Fairview beds were measured. Face samples for petrographic study and chemical analysis were obtained and a 1-1/2-ton sample was taken from the Fairview bed for washability tests at the Southern Experiment Station, Bureau of Mines, Tuscaloosa, Ala. The thickness of the beds and the physical conditions in and surrounding these beds were determined from cores of 24 diamond drill holes. The locations of these holes are shown in figure 4. Diamond-drill cores of the coal beds also were used for petrographic examination and chemical analysis.

The strata penetrated in drilling comprised the sandstones, shales, claystones, siltstones, clays and fourteen coal beds of the Pottsville formation.

13/ See footnote 10.

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The Fairview bed offers the best possibilities for future development. The reserve of coal in the Fairview bed, underlying two areas, is estimated at 14,580,000 tons, of which 11,660,000 tons should be recoverable (see fig. 4). Some areas of unmined coal may exist in the Hammond, Coal City, Broken Arrow, and Marion beds, but the reserves are small, and the areas are not favorable for large-scale mine development.

The slickensided nature of the coal and the folding of the bright bands (anthraxylon) show that the coal has been subjected to considerable contortion, probably due to folding and the resulting earth movement. The Fairview coal is classified, petrographically, as a bright coal containing a large percentage of anthraxylon.

The rank classification of the coals in the Coosa field is high-volatile A bituminous. The weighted average ash and sulfur content of samples of coal from the Fairview bed, as-received basis, are 14.0 and 3.8 percent, respectively. Analyses of sulfur forms in the Fairview coal show that about 80 percent of the total sulfur is pyritic, 19 percent organic, and 1 percent sulfate.

The as-mined ash and sulfur content of the sample obtained from the Soot Creek mine for washability studies was 12 percent ash and 3.6 percent sulfur. The results of the washability study indicate that washed coal up to 4-inch will contain approximately 10.1 percent ash and 2.47 percent sulfur if jigs and wet tables are used for cleaning. Assuming that the shale and rash partings in the mine are gobbed, a yield of about 83.5 percent of the washery feed should be obtained.

The Fairview and Coal City basins are parts of the north half of a composite structure known as the Coosa trough. Many approximately parallel transverse faults cross the bottoms of the basins. These transverse faults have not reduced the available tonnage of coal, but they have been detrimental to mining.

Minnesota Creek Area, Gunnison County, Colo.

Additional sources of coking coal west of the Continental Divide are needed for western steel plants. A reconnaissance of the Minnesota Creek area (about 6 miles east of Paonia) in Gunnison County, Colo., indicated large reserves of bituminous coal; and as regional movements have raised the rank of some western coals, it was believed that the bituminous coal in the Minnesota Creek area might have qualities suitable for the manufacture of metallurgical coke. This area was studied to determine whether the coals would produce coke of this quality.14/

Four diamond-drill holes to obtain 2-1/4-inch diameter cores and one hole to recover 8-inch cores for carbonization tests were drilled in this area (see fig. 5). The results of the investigation are summarized as follows:

At least four minable beds 4 feet or more thick and as many as 6 minable beds of high-volatile bituminous coal were penetrated in the drill holes. The area contains minable coal reserves of 781,000,000 tons, of which 164,900,000 tons are measured, 122,800,000 tons are indicated, and 493,300,000 tons are inferred. Assuming a recovery of 80 percent, 624,800,000 tons of coal should be recovered from the area. The weighted average moisture, ash, and B.t.u. values on an as-received basis for the cores from all minable beds (partings 3/8-inch or more thick removed) follows:

14/ See footnote 9.

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Figure 5. - Minnesota Creek coal area, Colo. (base map, U. S. Geol. Surv., Mt. Gunnison quadrangle.)

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Moisture, percent	10.0
Ash, percent	6.6
B.t.u.	12,121

The weighted average oxygen content (moisture-ash-free basis) for cores of all minable beds penetrated is 12.8 percent, whereas 10 percent is usually considered a maximum in metallurgical coking coals. The coal from one bed produced only enough coke for the tumbler test, which showed that this coal produced a coke inferior to coke made from the Lower Sunnyside coal bed in Utah.

Castleman Basin, Maryland

An investigation by diamond drilling in the Castleman Basin, Garrett County, Md., was completed. Forty holes were drilled in an area of about 14,000 acres in the center of this field south of Grantsville and U. S. Highway 40. Estimates of reserves and a final report giving the results of the investigation are in preparation.

Deep River Coal Field, North Carolina

Diamond drilling in the south part of the Deep River field was completed. Two deep diamond-drill holes were completed for a total of 4,682 feet of drilling. The Cumnock bed is the only important coal bed, from a mining standpoint, in this field. This bed was 47 inches thick in the first hole drilled and 41 inches thick in the second hole.

Coal Creek, Gunnison County, Colo.

An investigation by diamond drilling to determine minable reserves of coking coal in the Coal Creek district, Colorado, was completed. Twenty holes were diamond-drilled to obtain 2-1/4-inch-diameter coal cores, and two holes to obtain 8-inch cores for carbonizing tests. The investigation resulted in the development of a reserve of over one hundred million tons of coal, from which can be produced a metallurgical coke considerably stronger than the average coke made from coal from the Lower Sunnyside bed in Carbon County, Utah.

Study of Coking-Coal Reserves

An investigation was begun to determine the known recoverable reserves of coking coal in the United States. The field work consists in obtaining all available data from present and past mining operations, exploration conducted by owners of coal lands, operators of coal mines, and Federal and State agencies. Owners of coal lands and operators of coal mines are contacted to obtain, on a confidential basis, the following:

1. Map of the property.
2. Maps of mines, showing extent of workings.
3. Thickness of the coal beds and location of coal outcrops.
4. Maps showing locations of drill holes.
5. Logs of drill holes, giving bed sections and bed names.
6. Chemical analyses of the coals.
7. Life production of each mine.

Requests for this information have met with the full cooperation of these owners and operators.

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From the field data, work maps are prepared from which the average thickness of coal and the remaining coal reserves are computed. Where production for the life of a mine and the average thickness of the coal bed are known, the percentage of recovery from the bed is calculated. Tabulations of recoverable reserves are made by quadrangles and will be reported by counties or other geographical areas in such a manner that data given in confidence by the land owners and operating companies will not be revealed. Reserves are to be reported in two categories - measured (proved) and indicated (probable). In the Appalachian region, minable reserves are to be reported in the following thickness ranges: 14 to 28 inches, 28 to 42 inches, and over 42 inches.

The investigation was begun in the northern Appalachian region because of its importance as a supplier of coking coal, and the work to date has been concentrated in the medium- and low-volatile coal fields in central Pennsylvania, southern West Virginia, and eastern Kentucky.

The first field party of mining engineers began work in the Indiana quadrangle, Indiana County, Pa., in August 1948, and as personnel became available, additional parties were activated. At the end of the year, five field parties were collecting data - two in Pennsylvania, two in southern West Virginia, and one in eastern Kentucky. At the request of the National-Security-Resources Board, one of the field parties in Pennsylvania is working in the Connelsville and Uniontown areas of Fayette and Westmoreland Counties to determine the recoverable reserves of coking coal in these districts that would be available for continued operation of beehive coke ovens in the event of a national emergency.

Mining Methods and Practices

The Coal Industry of Brazil

For some years during and following the war, a vigorous program of technical improvement and production expansion in coal fields of southern Brazil was carried on by the United States Government and the Brazilian Government through the agency of Departmento da Producao Mineral.15/, 16/ This was initiated by the Foreign Economic Administration but subsequently was transferred to the Bureau of Mines.

As a result of concerted efforts in this area, the rate of production in the Santa Catarina field, which produces metallurgical fuel for the national steel industry, was raised from 268,000 tons in 1940 to 980,000 tons in 1946.

Production was increased substantially, also, in the important industrial coal field of northeastern Parana. This field was opened during the war and should become an important source of railway locomotive fuel and general-purpose fuel for the Sao Paulo industrial area.

Technologic improvements included the introduction of pneumatic mining equipment in the Santa Catarina mining field, installation of a modern central preparation plant, marked improvement in railway facilities, and the installation of improved port facilities at Imbituba and Laguna at the Atlantic coast terminal of the Dona Tereza Cristina Railway.

15/ Good, John E., Abreu, Alvaro, and Fraser, Thomas, The Coal Industry of Brazil. Part I. General Economy, Production, and Marketing: Bureau of Mines Tech. Paper 713, 1949, 38 pp.

16/ Fraser, Thomas, Amostragem e flutuacao de carvoes em liquidos pesadoes: Brazil Dept. Nacional da Producao Mineral, Avulso 7, 1947, 59 pp.

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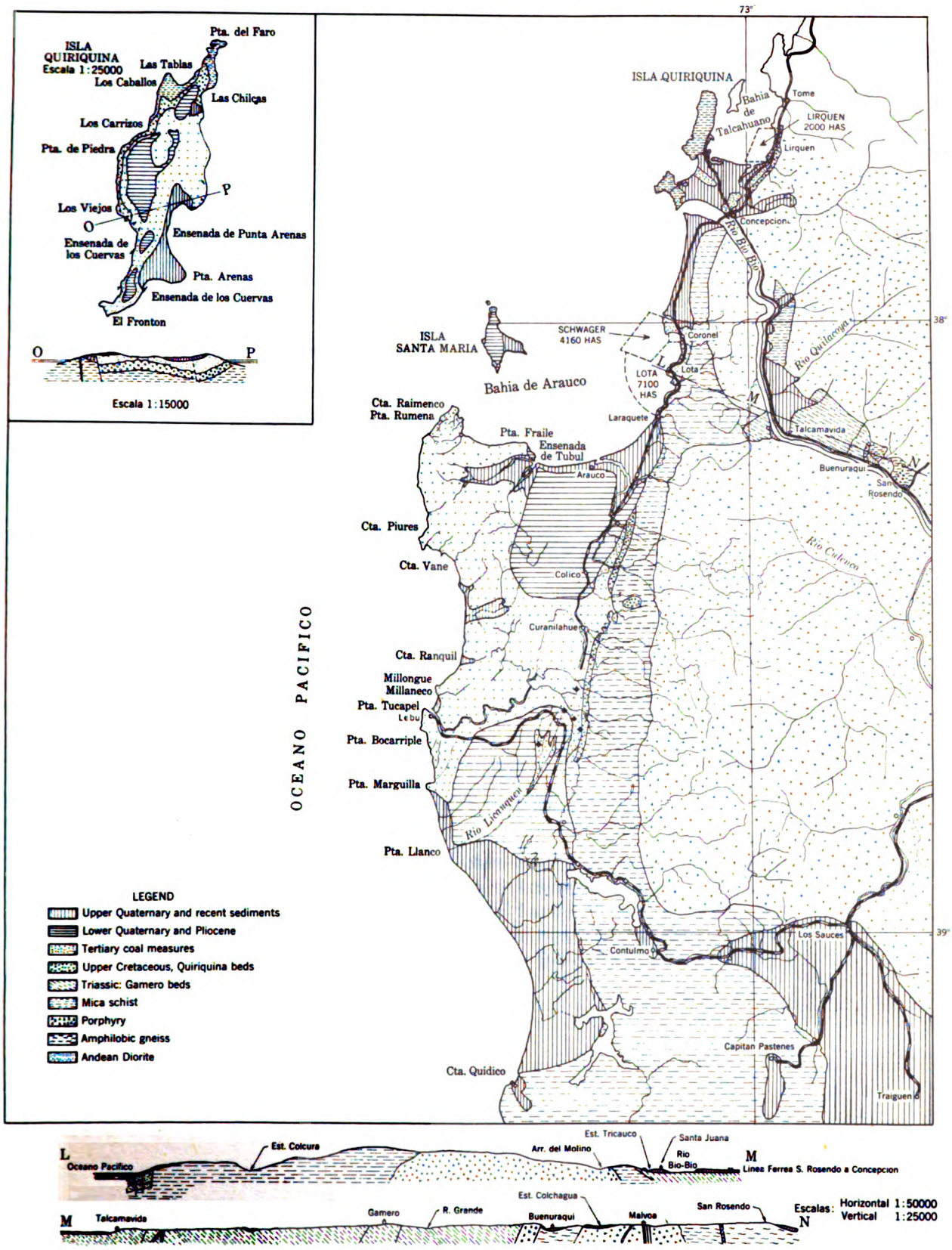

Figure 6. - Geologic map of the Arauco region, Chile.

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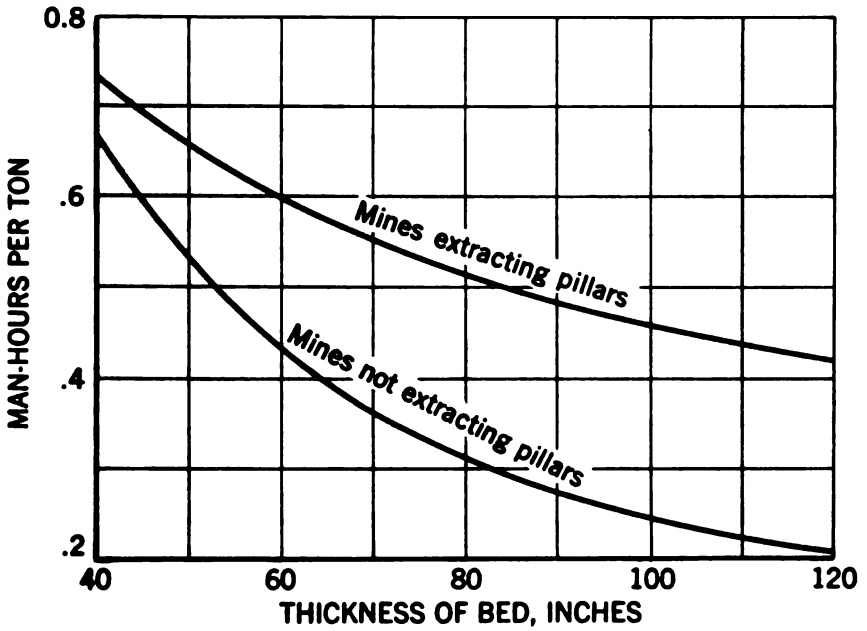

Figure 7. - Relation of man-hours per ton on mechanized units to thickness of bed. Pillars extracted and pillars not extracted. Mobile loading machines. (Man-hours computed from full 7-, 8-, or 9-hour shifts.)

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Coals of Chile

The results of an investigation of coals of Chile made at the request of the Chilean Government was published.17/ The investigation was made to determine whether the coal from beds mined in the Schwager and Lota mines will yield metallurgical coke suitable for use in the blast furnaces at the proposed steel plant at Concepcion; and whether the quality of the lower-rank coals from other areas in Chile, especially those in Magallanes, where the largest coal reserves of Chile appear to exist, can be improved by processing. Mining methods were studied, and decreased costs and increased production were recommended.

The principal coal-producing areas of Chile are adjacent to the Bay of Arauco, approximately 20 miles (32 kilometers) south of Concepcion (see fig. 6). The two principal mines are Lota and Schwager, which produce a high-volatile A bituminous coal equaling approximately 80 percent of the total Chilean output.

Mining practices at the Schwager and Lota mines are governed principally by the frequency and magnitude of the faults that displace the coal beds. The situation of the mine workings under the ocean and the ever-increasing distance of the advancing mining face from the mine openings create a problem of ventilation and transportation. The Schwager has begun to sink two new shafts 3,000 feet and will drive rock gangways to intercept the coal beds at approximately 4 miles from the shaft. These gangways will eventually be extended to 8 miles.

The coal-mining problem in Chile is not only physical but economic. There are coal regions in the United States where physical conditions are as adverse as those in some mines in Chile, but conditions in this country have been studied thoroughly, and efficient mining practices have been developed. Wages in the United States far exceed those paid for similar work in Chile, but on a dollar basis the labor cost per ton is materially less in the United States. This is due principally to the high ratio of non-producers to producers at most Chilean mines. The ratio is out of balance in most of these mines, and this results in a low over-all output per man. This condition probably developed from the theory that "many hands make light work", in years past when the wage scales were low; but today wage scales are high in comparison, and the resultant labor cost per ton is high. With fewer men for a required output, mechanization is not always possible, and it will be necessary to increase efficiency, either through mechanization or improved mining practices.

Extraction of Pillars with Mechanized Equipment

A progress report discussing the results of studies of pillar extraction with mechanized equipment at 10 mines in Pennsylvania, West Virginia, and Wyoming was issued.18/ The operational curves contained in the report and the conclusions reached are based upon detailed studies of mining operations at 55 coal mines in all of the important coal fields in the United States.

The relation of man-hours per ton (on mechanized units using mobile loading machines) to thickness of coal bed at mines practicing pillar extraction and at mines where pillars are not extracted is shown in figure 7. Man-hours per ton at the face

17/ Toenges, Albert L., Kelly, Leon W., Davis, J. D., Reynolds, D. A., Fraser, Thomas, Crentz, W. L., and Abernethy, R. F., Coals of Chile: Bureau of Mines Bull. 478, 1948, 106 pp.

18/ Turnbull, Louis A., and Toenges, Albert L., Mechanical Mining in Some Bituminous Coal Mines. Progress Report 5. Extraction of Pillars with Mechanized Equipment: Bureau of Mines Inf. Circ. 7527, 1949, 59 pp.

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are higher at mines where pillars are extracted than where pillars are not extracted. The difference in man-hours per ton increases as the thickness of the coal bed increases and ranges from 0.09 man-hour per ton for a bed 42 inches thick to 0.21 man-hour per ton for a bed 120 inches thick. Roof control generally is a greater problem where pillars are extracted, and the difference in man-hours per ton shown on the curves is due to additional time spent in timbering to protect the roof in operating sections. More men are required to set timbers where beds are thick than where they are thin.

Figure 8 shows the relation of man-hours per ton for total underground labor (using mobile loading machines) to thickness of coal bed at mines practicing pillar extraction and at mines where pillars are not extracted. Total labor employed underground ranges from 0.22 to 0.25 man-hour per ton more at mines where pillars are extracted than where pillars are not extracted.

Figure 9 shows the relation of tons of raw coal loaded per mobile loading unit per hour to thickness of coal bed at mines practicing pillar extraction and at mines where pillars are not extracted. This shows the small difference in coal loaded per hour in pillar extraction and where pillars are not extracted. This difference is about 2 tons per hour for all bed thicknesses. For example, in a 72-inch bed, production per loading machine per hour where pillars are extracted will be about 7 percent less than in mines where pillars are not recovered.

The most difficult problem encountered in extracting pillars with mechanized equipment is the effective control of not only the immediate roof but the entire overlying strata. The speed and regularity with which pillars are extracted help to obtain regular breaks in overlying strata along established pillar lines, thus relieving the weight at the coal faces. Many mines take advantage of natural cleats in roof rock to obtain frequent and systematic roof falls.

The production per mobile loading machine is 6 to 10 percent less in pillar mining than where pillars are not recovered. Production per conveyor per hour seems to be about the same in pillar and nonpillar mining.

Extraction of pillars in coal mines brings considerations of costs and effects on other property, which should be examined prior to adopting a plan for extraction.19/ Consideration should be given to effect upon coal beds above and below that in which pillars are to be extracted and to effects upon the surface that will result from the inevitable subsidence.

Coal Mining Methods and Practices in Western Arkansas

A preliminary study of mining methods and practices was made at six Arkansas coal mines - two in each of the coal areas.20/

The western Arkansas coal fields lie in the Arkansas River Valley region, which is bounded on the north by the Boston Mountains and on the south by the Ouachita Mountains. Erosion of folded structures has divided the coal area into separate mining districts (see fig. 10).

19/ Greenwald, H. P., Surface Factors Affecting Pillar Recovery: Mining Cong. Jour., vol. 35, 1949, pp. 54-57.

20/ Toenges, Albert L., and Fish, Edward L., Coal Mining Methods and Practices in Western Arkansas. Preliminary Investigation: Bureau of Mines Rept. of Investigations 4448, 1949, 25 pp.

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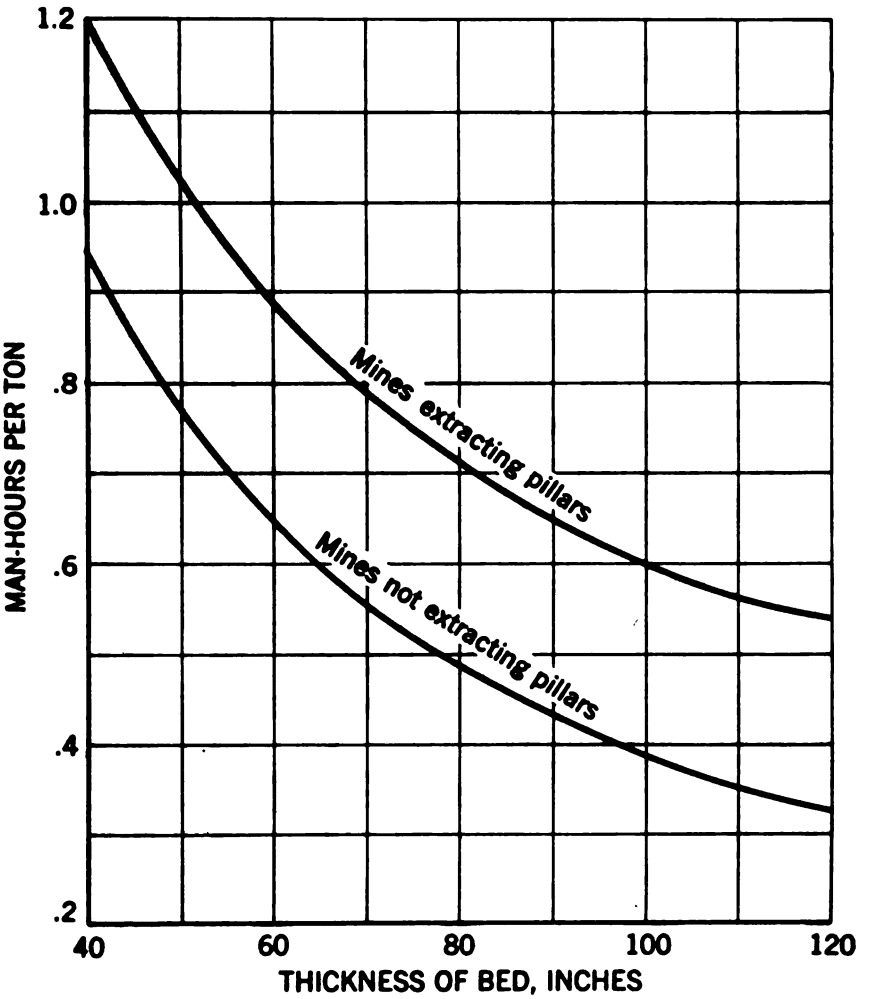

Figure 8. - Relation of man-hours per ton from total underground labor to thickness of bed. Pillars extracted and pillars not extracted. Mobile loading machines. (Man-hours computed from full 7-, 8-, or 9-hour shifts.)

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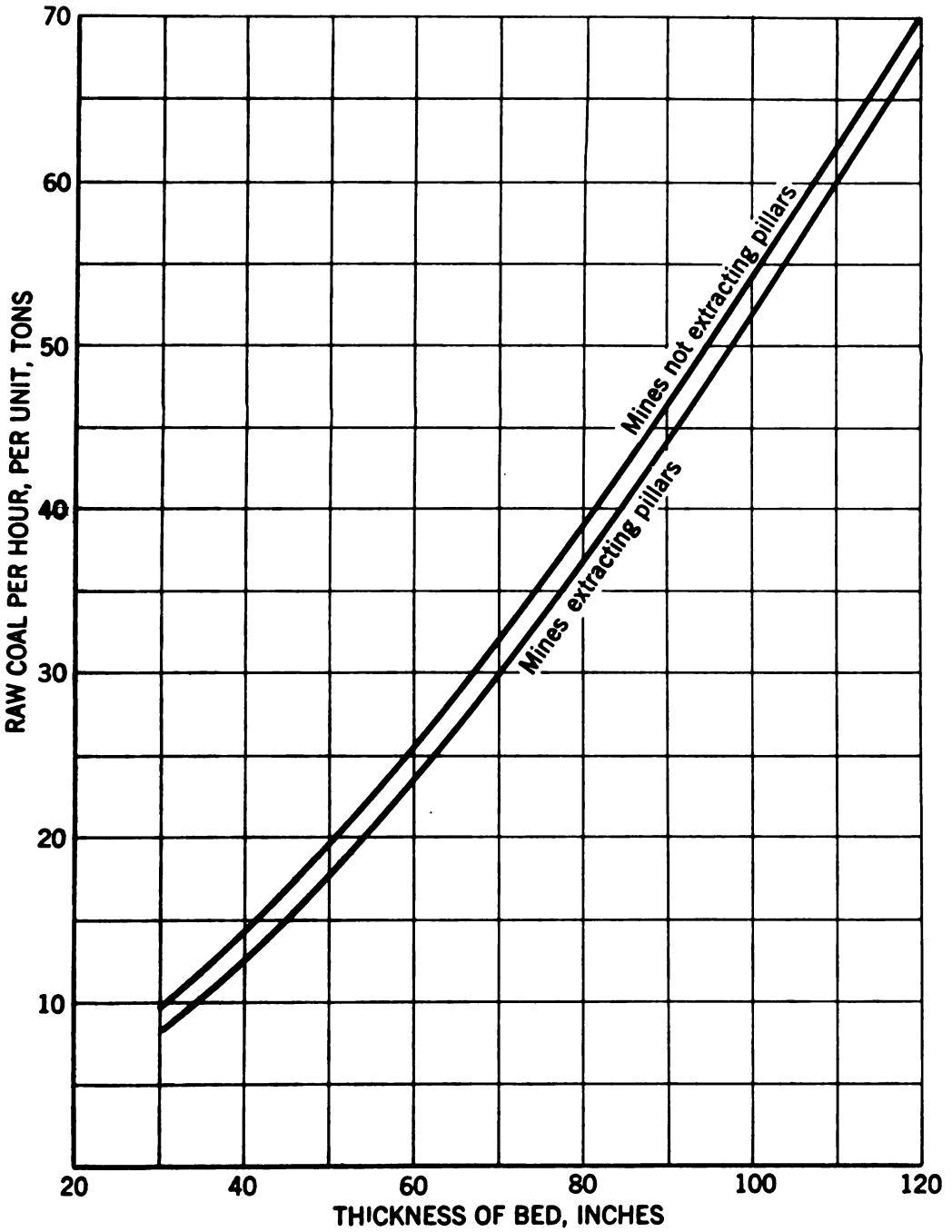

Figure 9. - Relation of tons of raw coal loaded per mobile loading unit per hour to thickness of coal bed. Pillars extracted and pillars not extracted. (Tons per hour computed from full 7-, 8-, or 9-hour shifts.)

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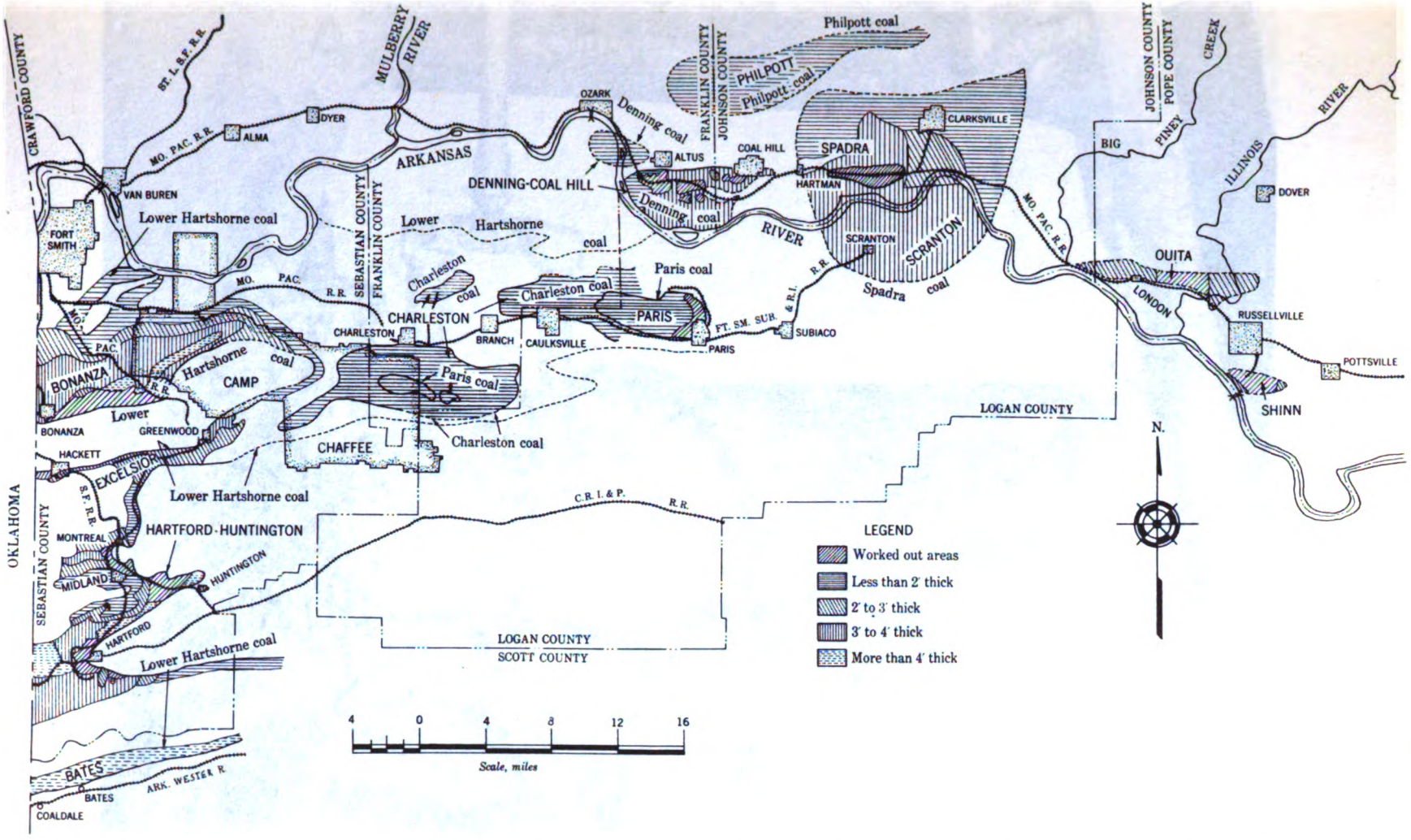

Figure 10. - Coal fields of western Arkansas. Compiled from unpublished map of western Arkansas coal fields. Arkansas Geological Survey, 1941.

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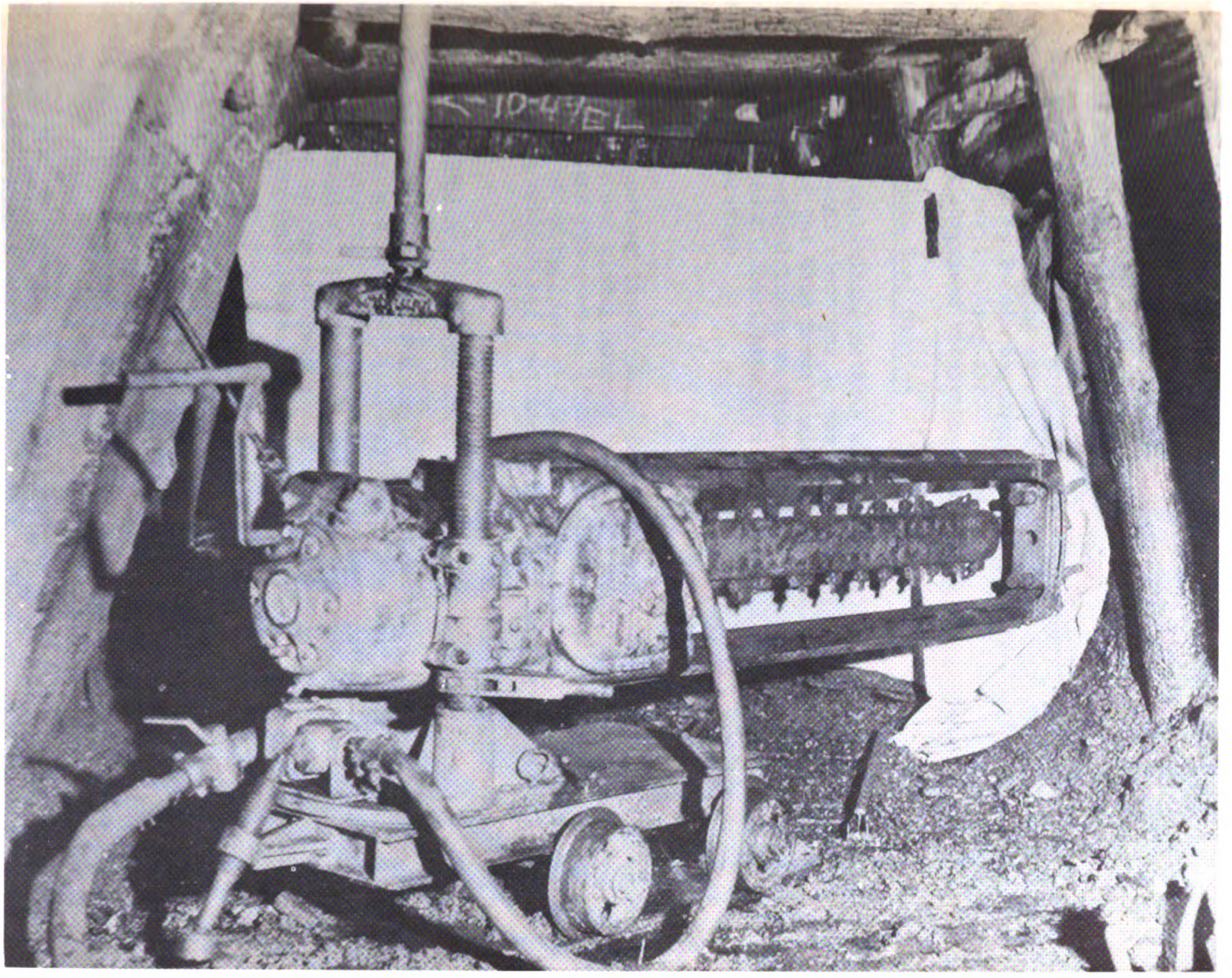

Figure 11. - Korfmann Universal shearing machine, Model SK 20, used in driving a 420-foot pillar road in a 28-foot coal bed without shattering the pillar.

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New and broader markets are needed for Arkansas coals. Larger markets would necessitate a longer work year, which could attract the skilled type workman not now available to the mines of this State. A market for smaller sizes than are now demanded by consumers of Arkansas coal also is needed, as further mechanization of the mines would result in degradation of the friable coals of this area. Present market demands are for large lump coal.

Slope Sinking Through Water-Bearing Strata

The Bell & Zoller Coal & Mining Co. recently completed the construction of two slopes through water-bearing strata at mine 3, near Ziegler, Ill. These operations, which were studied by Bureau engineers,21/ were in the Big Muddy River Valley, and construction was particularly difficult because the strata from the surface to a depth of 70 feet comprised water-saturated, sandy clay, and sand.

Missouri River Basin Coal Studies

Studies of coal-mining methods and practices, electrical-power requirements at coal mines, and coal resources in the Missouri River Basin were continued. Detailed investigations in the Wind River Region, Fremont County, Wyo., were completed. A field study of coal-mining operations and coal resources in that part of the Kansas coal fields in the Missouri River drainage area was completed, and a report of this work is in preparation.

Greek Lignite

At the request of the Economic Cooperation Administration, an investigation of lignite deposits and mining methods and practices in Greece was made to determine whether sufficient reserves of lignite (the only fuel in Greece) were present for the development of mines to produce 1,000,000 tons annually, and to recommend improvements in mining methods and practices that would increase production and reduce costs.

Anthracite Mechanical Mining Research

The Bureau's participation in anthracite mechanical-mining studies has consisted in providing new and untried machinery22/ and engineering services. Cooperating companies have provided underground test sections, power, labor, supplies, and engineering services. Projects are active in the northern, western, middle, and southern fields of the anthracite region of Pennsylvania. Headquarters for activities are at Schuylkill Haven, Pa.

Air-powered German lightweight shearing machines have been introduced by the Bureau for both flat and pitching beds. In the flat beds of the northern field, a project was completed for driving a pillar road 420 feet long in a 28-foot-thick coal bed without shattering the pillar. This project was undertaken in connection with changing the mining system in an area containing 1,100,000 tons of marketable coal in pillars. The type of machine used for this work is shown in figure 11.

21/ Shields, Joseph J., and Turnbull, Louis A., Slope Sinking Through Water-Bearing Strata at Mine 3, Bell & Zoller Coal & Mining Co., Williamson County, Illinois: Bureau of Mines Inf. Circ. 7509, 1949, 5 pp.

22/ Buch, John W., Trends in Anthracite Underground Mine Mechanization: Trans. 7th Ann. Anthracite Conf., Lehigh University, Bethlehem, Pa., May 1949, pp. 195-206.

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In the southern field, work has been completed that establishes the performance of lightweight air-powered shearers as a fourfold improvement over hand methods in developing thin coal beds on heavy pitch. One of these machines is illustrated in figure 12.

In connection with studies of mine development, conventional timbering methods were found to be wasteful of time and probably of material. In order to design a fully mechanized timbering method, tests were undertaken to establish data for loads borne by roadway supports. Preliminary laboratory test work is nearly completed. This work will be used as the basis for designing and building an experimental telescopic shield to improve efficiency and safety for the face worker and to eliminate the arduous labor of conventional timbering methods.

Hydraulic back filling and hand packing have been practiced for a long time in the anthracite area, and some small amount of work has been done pneumatically. However, all such work has proved so costly that full advantage has not been taken of back filling for roof control and pillar strengthening. Two years ago the Bureau imported, for experimental purposes, a packing machine of German origin that has had widespread use on the Continent and in England for many years. This machine was installed on the surface at a Northern field mining operation for preliminary test runs that proved very satisfactory. The machine has been installed underground for further testing. The machine is shown in figure 13.

A vibrating-blade coal planer patterned in principle after those experimented with in Germany during the years of World War II was designed by and is now being built for the Bureau.

Studies on the Use of Diesel Engines Underground

Work has been completed on the removal of aldehydes from Diesel exhaust gas by scrubbing with an aqueous solution containing sodium sulfite (10 percent by weight) and hydroquinone (0.5 percent by weight).23/

A study of the variables that affect the average rate of reaction during inflammation in a Diesel engine was begun. Under the heterogeneous conditions existing during combustion in such an engine, it is difficult to separate the effects of different variables. However, preliminary results indicate that the following relationships exist: (1) Fuel concentration is of primary importance in determining the average rate of reaction. (2) The reaction rate increases with the increased density of the intake air; as previous work has established that reaction rate is independent of the partial pressure of oxygen, this effect is believed to be due to the better atomization and distribution of the fuel obtained with greater air density.

The lower limit of inflammability of natural gas (the concentration at which essentially all of the gas is burned) in a CFR Diesel engine was determined to be 4.8 percent in the intake at a liquid fuel:air ratio of 0.0066 pound per pound. Natural gas alone could not be ignited by compression in the CFR Diesel engine. The lower-limit flammability of hydrogen in a 4-cylinder Hercules DOOC Diesel engine was similarly determined to be 12 percent in the intake at a minimum liquid fuel:air ratio.

23/ Davis, R. F., and Elliott, M. A., The Removal of Aldehydes from Diesel Exhaust Gas: Trans. Am. Soc. Mech. Eng., vol. 70, 1948, pp. 745-750.

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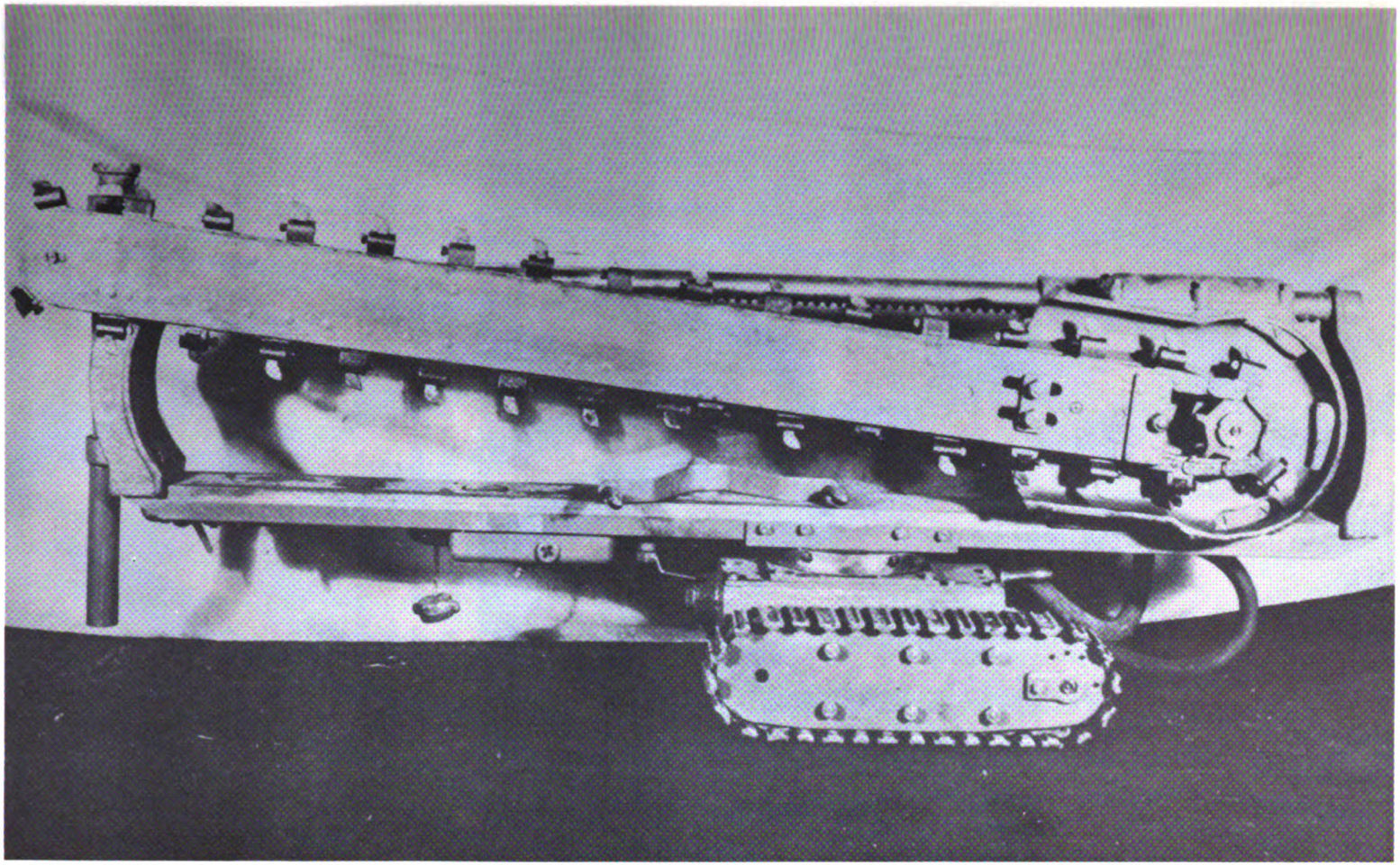

Figure 12. - Korfmann shearing machine, Model SK 15, used in establishing performance data for thin, steeply pitching beds of the Southern field.

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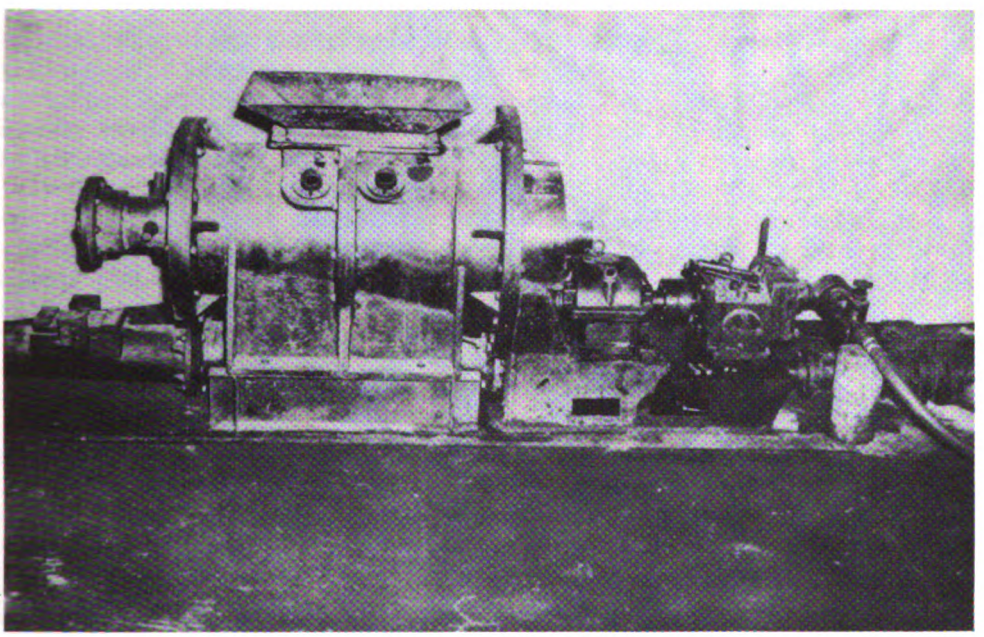

Figure 13. - Brieden pneumatic packing machine. Rock for building pack introduced at top of machine and fed to air stream at bottom by a segmented, rotary, internal drum powered by an air motor. Performance data not available.

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The results of studies made by the Bureau of Mines on the use of Diesel engines underground were presented.24/ These studies, as well as those made by other agencies, have shown that Diesel engines can be operated safely underground if the following conditions are met:

1. Ventilation must be provided to dilute adequately and remove from the underground atmosphere all harmful and objectionable constituents (carbon monoxide, oxides of nitrogen, carbon dioxide, oxides of sulfur, aldehydes, and unburned carbon) of the exhaust from the engine.

2. Flame protective devices must be provided for the intake and exhaust systems of the engine to prevent ignition of a flammable atmosphere.

3. Fire hazards in handling Diesel fuel underground must be eliminated.

Under normal operating conditions with engines in proper mechanical condition, the concentration of flammable constituents in the exhaust is low enough to permit their dilution to a safe value in the underground atmosphere by supplying adequate ventilation. To avoid undesirable operating conditions, careful attention must be given to the maintenance and adjustment of Diesel engines in underground service.

Tests of three different Diesel engines have shown that a flammable atmosphere surrounding the engine could be ignited by flame issuing from either the intake or exhaust systems. Flame arrestors should therefore be provided for the intake and exhaust openings.

Flame arrestors on Diesel engines may operate at elevated temperatures, depending upon their position in the exhaust systems.

Upon the basis of information obtained in the testing station and in the field, the Bureau of Mines has issued schedules 25/, 26/ outlining the procedure for testing Diesel mine locomotives for permissibility and making recommendations for use of Diesel engines underground.

International Conference of Directors of Mine Safety Research

The Fifth International Conference of Directors of Mine Safety Research was sponsored by the Bureau of Mines and held in Pittsburgh on September 20 to 25, 1948. The Conference was attended by official delegates from five European countries and the United States and by many guests from these and other countries. The technical sessions were devoted to presentation and discussion of recently developed experimental data on explosives, firedamp, coal-dust explosions, mine fires, electricity and haulage, and health and safety in mining.

24/ Elliott, Martin A., Review of Bureau of Mines Work on the Use of Diesel Engines Underground: Bureau of Mines Rept. of Investigations 4381, 1948, 28 pp.

25/ Bureau of Mines Schedule 22 - Procedure for Testing Diesel Mine Locomotives for Permissibility and Recommendations on the use of Diesel Locomotives Underground: Federal Register, vol. 9, No. 230, Nov. 17, 1944, pp. 13742-13753, incl.

26/ Bureau of Mines Schedule 24 - Procedure for Testing Mobile Diesel Powered Equipment for Non-Coal Mines: Federal Register, vol. 14, No. 67, April 8, 1949, pp. 1671-1677, incl. Correction for Schedule 24: Federal Register, vol. 14, No. 113, June 14, 1949, p. 3201.

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Toxic Mine Atmospheres

In connection with the work carried on under the provisions of the Federal Coal Mine Inspection Act, approximately 15,000 samples of mine air were analyzed to determine the adequacy of ventilation in coal mines and to aid in the control and extinguishment of mine fires. Seven hundred and fifty samples of air-borne dusts and other mineral materials were examined to determine the free-silica content and dust concentration in connection with studies relating to health hazards from dust in mining operations. These analytical operations required more than 100,000 individual determinations.

As the sampling and analysis of mine atmospheres play an important part in promoting and maintaining safe and healthful conditions in mining, information on the subject was revised and brought up to date in a publication designed as a practical working manual for the mine operator and mining engineer.27/

An investigation of the causes of explosions and formation of carbon monoxide in compressed-air systems demonstrated that a "carbon-oxygen complex" exists in the carbon deposits that are formed normally in such systems.28/ This complex may act as an initiator of the exothermic reactions that result in explosions and formation of carbon monoxide in compressed-air systems. The findings of this investigation may point the way to increased safety in the use of compressed-air in mining and other industries.

Approval testing was continued on respiratory protective devices applicable to use in the mining and mineral industries. Six new approvals and 37 extensions of approval, representing modifications and improvements in design, were granted. To aid in the selection of respiratory protective devices, and to bring up to date information on the subject, a listing of the newly approved devices was prepared.29/

Electrical Equipment for Mines

Unless electric equipment is built, maintained, and used properly in gassy coal mines, such equipment can be hazardous from the standpoint of initiating mine fires and explosions. As a means of avoiding this hazard, the Bureau of Mines has established certain standards, published as "schedules," for the guidance of manufacturers who produce mine equipment. A design of equipment that passes the tests and inspections prescribed by these standards is formally approved by the Bureau of Mines and becomes known as "permissible" equipment, that is, permissible for use in gassy coal mines. Each item of equipment of this design sold by the manufacturer as permissible is identified by an approval plate bearing the Bureau of Mines' seal. Manufacturers in increasing numbers, both in this country and abroad, are becoming more and more interested in the standards of the United States, so much so that it was necessary to reprint Schedule 2E 30/ to satisfy the large demand for this publication.

27/ Berger, L. B., and Schrenk, H. H., Sampling and Analysis of Mine Atmospheres: Bureau of Mines Miners Circ. 34, 1948 revision, 103 pp.

28/ Busch, H. W., Berger, L. B., and Schrenk, H. H., The Carbon-Oxygen Complex as a Possible Initiator of Explosions and Formation of Carbon Monoxide in Compressed-Air Systems: Bureau of Mines Rept. of Investigations 4465, 1949, 22 pp.

29/ Berger, L. B., Supplemental List of Respiratory Protective Devices Approved by the Bureau of Mines (additions to list published in Inf. Circ. 7444): Bureau of Mines Inf. Circ. 7513, 1949, 3 pp.

30/ Bureau of Mines Schedule 2E - Procedure for Testing Junction Boxes and Electric Motor-Driven Mine Equipment for Permissibility: Federal Register, vol. 10, No. 41, Feb. 27, 1945, pp. 2230-2240, incl. Amendment to Schedule 2E: Federal Register, vol. 12, No. 219, Nov. 7, 1947, pp. 7285-7287, incl.

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Under the eight schedules now in effect, a manufacturer can have almost every type of equipment used at or near the face of active mine workings investigated for its liability to ignite gas or coal dust or a combination of these. During the fiscal year 1949, 53 approvals were issued. These included the designs for 21 loading machines and conveyors, 1 cutting machine, 1 continuous miner, 5 drilling machines, 1 mining-machine truck, 2 air compressors, 1 battery-operated shuttle car, 1 post puller, 1 timbering machine, 5 fan-drive units, 1 greasing truck, 2 battery-operated utility trucks, 1 car-spotting hoist, 1 junction box, 1 distribution box, 1 cleaner, 3 rock-dust distributors, 1 ten-shot blasting unit, 1 one-shot blasting unit, 1 electric cap lamp, and 1 flame safety lamp.

Electrical Control Mechanisms in Underground Pumping Plants

The physical lay-out of two large, modern, underground, automatic pumping plants in the anthracite region are described.31/ A detailed technical explanation, accompanied by wiring diagrams, was given of the functioning of the individual electrical control mechanisms and safety features incorporated into the controls and the effect each mechanism has on the over-all automatic operation of the plant.

Roof Control

Falls of roof and coal account for about half the total fatalities in coal mines. Bureau of Mines engineers have conducted studies of roof-control measures through the years when funds for such work were available, and some practical solutions to special problems, such as "bumps," have been developed. However, no new roof-control method that could be applied generally in mines was discovered until 1948, when two Bureau of Mines engineers investigated a roof-control method used for many years in metal mines in southeast Missouri, recognized its possibilities, and applied the method in coal mines and some ore mines in the United States. Experimental work was conducted to prove its value and practicability under actual operating conditions.32/, 33/, 34/ The method is referred to as "Suspension Roof Supports."

The principle involved in this improved method of roof control is the prevention of initial sag and subsequent failure of thin bands of roof strata by reinforcing the strata with steel rods as soon as the roof is exposed. These supports eliminate the use of timber sets and posts, and are of especial value in mechanized mines where dislodgment of the standard supports is likely to result in roof failure and possible injury to those persons in the immediate vicinity.

During the fiscal year, in answer to numerous requests by coal companies, examinations were made of the roof and mining conditions at various mines by personnel of the roof-control group to determine if suspension roof supports were applicable to the local conditions. These examinations and recommendations were made with respect to the installation of supports. During the fiscal year, 115 mine examinations for this purpose were made in 11 States.

31/ Durham, A. C., Design and Operation of Electrical Control Mechanisms in Underground Pumping Plants: Bureau of Mines Inf. Circ. 7476, 1948, 21 pp.

32/ Thomas, E., Seeling, C., Perz, F., and Hanson, M., Control of Roof and Prevention of Accidents from Falls of Rock and Coal: Bureau of Mines Inf. Circ. 7471, 1948, 9 pp.

33/ Thomas, E., Suspension Roof Supports: Coal Age, vol. 53, 1948, pp. 87-91.

34/ Thomas, E., Conventional Timbering vs. Suspension Supports: Mechanization, vol. 13, 1949, pp. 147-152.

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It is known that suspension roof supports have been installed in 60 bituminous-coal mines and 8 iron-ore mines situated in 8 States, and it is estimated that 147,038 feet of mine passageways are now supported by this means.

During the fiscal year, a stratascope has been developed and is near completion at the Naval Gun Factory, Washington, D. C. This instrument will provide precision and accuracy in determining strata separation, which usually precede failure, by exploring boreholes drilled in thinly bedded mine-roof rocks.

A "torquometer" was obtained near the close of the fiscal year, and a limited number of readings of bolt tensioning was obtained at two installations where suspension roof supports were installed, but the data are insufficient to permit accurate interpretation. To supplement the readings obtained in the field, a schedule of laboratory tests has been set up to determine factors that may affect ultimate results. Indications are that the "torquometer" readings will provide significant information relating to effectiveness of suspension-rod anchorages.

Anthracite Flood Prevention

Investigations of underground water pools, the "buried valley" of the Susquehanna River, anthracite reserves, existent pumping facilities, existent drainage tunnels, and a study of deep-well pumps and deep-well-pumping installations in the anthracite region were conducted in the search for a solution to the anthracite flood-prevention problem. The investigation of underground water pools was completed.35/ The survey revealed that there are 91 billion gallons of water impounded in 159 pools in underground workings and 2.3 billion gallons impounded in abandoned stripping excavations.

One phase of the mine-water problem, as it pertains to the northern field of the anthracite region, is the presence of the buried valley (filled ancient channels) of the Susquehanna River. The present river channel is above the channels of the ancient waterways, which the north branch of the Susquehanna River has filled with clay, sand, and gravel deposits. This valley fill is water-bearing and irregular in trend and depth. The thickness of the depositional material generally is 100 to 150 feet, though in places it is more than 300 feet thick. The coal measures underlie and outcrop beneath and at the sides of these deposits. A vast tonnage of anthracite has been mined from the area beneath the buried valley, and, because a large tonnage remains unmined, it is of prime importance that present and future mine operations in this area know the configuration, extent, and nature of the water-bearing deposits, and the relative position of the underground workings to them to prevent the mine workings breaking into the valley-fill deposits unexpectedly, with resultant loss of life and property.

To date, information on 13,000 boreholes has been compiled, and from this and other information, 57 contour maps have been prepared.

A study was begun to determine possible sites for drainage tunnels in the northern and southern fields.

35/ Ash, S. H., Eaton, W. L., Hughes, Karl, Romischer, W. M., and Westfield, J., Water Pools in Pennsylvania Anthracite Mines: Bureau of Mines Tech. Paper 727, 1949, 78 pp.

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Coal Washing

Cleaning and Recovery of Fine Coal

The second commercial installation of the "kerosine-flotation" process was made at a mine in the Birmingham, Ala., district, where it operates on minus 10-mesh fines screened from run-of-mine coal.

This plant is providing a solution to what has always been a difficult problem in wet-preparation plants - the recovery, cleaning, and dewatering of "fines." The first installation of the kerosine-flotation process, reported last year, is being used for treatment of the underflow sludge from dewatering screens. Both plants are operating successfully for the recovery of coking coal. They have been built as a result of Bureau experimental plant and laboratory work initiated several years ago.

Laboratory tests have been made of the application of this process to the cleaning of middling products from jig and table plants. The results show that the development of the process will make additional recovery of coking coal feasible from seams that are high in ash because of their high bone-coal content.

Coal Washery-Water Clarification

Under a cooperative agreement with the Truax-Traer Coal Co., Chicago, Ill., the Bureau of Mines installed and operated a 14-inch-diameter cyclone thickener at the preparation plant of the Shamrock Mine, Kayford, West Virginia. The unit was placed in the fine-coal washery circuit and used as a water-clarification device. Test data indicate the recovery of substantially all coal particles 100-mesh and larger in size. Less efficient recovery is experienced as the particle size decreases with but a one-third recovery of these solids finer than 325-mesh.

The rate of flow through the unit and the inlet nozzle pressures were controlled by adjusting the speed of a 6- by 4-inch centrifugal pump driven by a 25-hp. motor. The underflow spigot had a fixed diameter of 1-3/8 inches, and the inlet-feed nozzle was fixed at 2 inches inside diameter. The overflow nozzle was removable and interchangeable. Tests with 2-5/8-inch and 2-3/8-inch overflow orifices gave results that might be considered practical for thickener performance, with a thickener underflow product of 50 percent or less water and a recovery of about one-half the solids from the washery circuit.

This one cyclone recovered 5 to 6 tons per hour of marketable coal, which was previously lost. The demonstration of the usefulness of this device in the preparation plant influenced the cooperating company to install three additional cyclones to handle the entire water volume of the fine-coal washery circuit.

Washing Characteristics of Chilean Coals

At the request of the Chilean Government, the washing characteristics of Chilean coals were studied. Coal samples were collected from 7 mines operating in 11 beds, and the test data were reported.36/ Many of the beds may be washed at high-gravity with the simplest of modern coal-washing equipment and yield a high-grade, low-ash, low-sulfur product. The sulfur content of all samples was either very low (1 percent or less) or exceeded 3 percent. Where the sulfur content was high, little reduction in sulfur content could be expected by mechanical cleaning.

36/ See footnote 17.

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Coal samples from the Province of Concepcion responded well to mechanical cleaning for ash reduction, producing a float-coal ranging from 2.2 to 6.7 percent in ash content. The Province of Voldivia coals, containing less than 10 percent ash in the raw state, did not respond to further ash reduction. The coals from the Province of Magallanes, although very low in sulfur, contained about 17 percent ash. The samples showed very little free rock, and examination of the washability charts indicated that the coals are not adapted to substantial improvement by washing, except by an uneconomic sacrifice in yield.

Coal Washing in Washington, Oregon, and Alaska

A general summary of the status of coal-washing practices in Washington, Oregon, and Alaska - an area in which some of the coals mined are more difficult to wash than those mined elsewhere in the country - has been published.37/ Many of the coal beds mined in Washington and Alaska contain more impurities than those mined elsewhere, and this circumstance contributed to the early interest in mechanical cleaning. A much more important factor, however, was the inclination of the coal beds. With steeply pitching beds, hand sorting by the miner at the face is impossible; consequently, all material must be loaded and dealt with on the surface, just as is now proving the case with mechanical mining of flat beds. Thus, "full-seam" mining afforded the same stimulus to the early development of coal washing in the Pacific Northwest that it is providing under mechanization in the rest of the country today.

Washington leads all other States in the percentage of its total production that is cleaned mechanically - 82.1 percent, in comparison with the national average of 25.6 percent in 1945. The washing problems in this State are highly variable, because the mountain-building forces that created the Cascade Range caused such intense folding and faulting of the beds in some fields that the rank of the coal was increased to anthracite, whereas in other fields, more distant from the mountains, the beds were relatively undisturbed, and the rank of the coal ranges down through subbituminous to lignite. The washability of several Washington coals is illustrated by figures 14 and 15.

Coal occurs at a number of locations in western Oregon, but mining has been limited principally to the Coos Bay field on the southwestern coast of Oregon. The Southport mine of the Coast Fuel Corp. requires a washing plant to remove the interbedded clay of the Beaver Hill bed. This plant incorporates provision for screening the run-of-mine product at about 4-inch size, hand-picking the lump, and optionally crushing the clean lump to join the screenings as feed for a Forrester-type jig. A specific-gravity analysis of the raw coal from this mine is given in table 4.

Coal production in Alaska during recent years has been divided about equally between the subbituminous coals of the Nenana field and the bituminous coals of the Matanuska Valley. The subbituminous coals, mined at Suntrana, are clean enough to be prepared by screening and some handpicking. In contrast, the coals of the Matanuska Valley contain more impurity than most so-called "dirty" coals mined in the United States and consequently must be washed to render them suitable for even the Alaska market. The proportion of washery feed rejected as refuse in Alaska averaged 33 percent in 1945, a far higher figure than was recorded for any State in the country.

37/ Geer, M. R., and Yancey, H. F., Coal Washing in Washington, Oregon, and Alaska: Am. Inst. Min. and Met. Eng. Tech. Pub. 2566F, Mining Trans., vol. 108, 1949, pp. 200-204; Mining Eng., vol. 1, No. 6, 1949, pp. 200-214.

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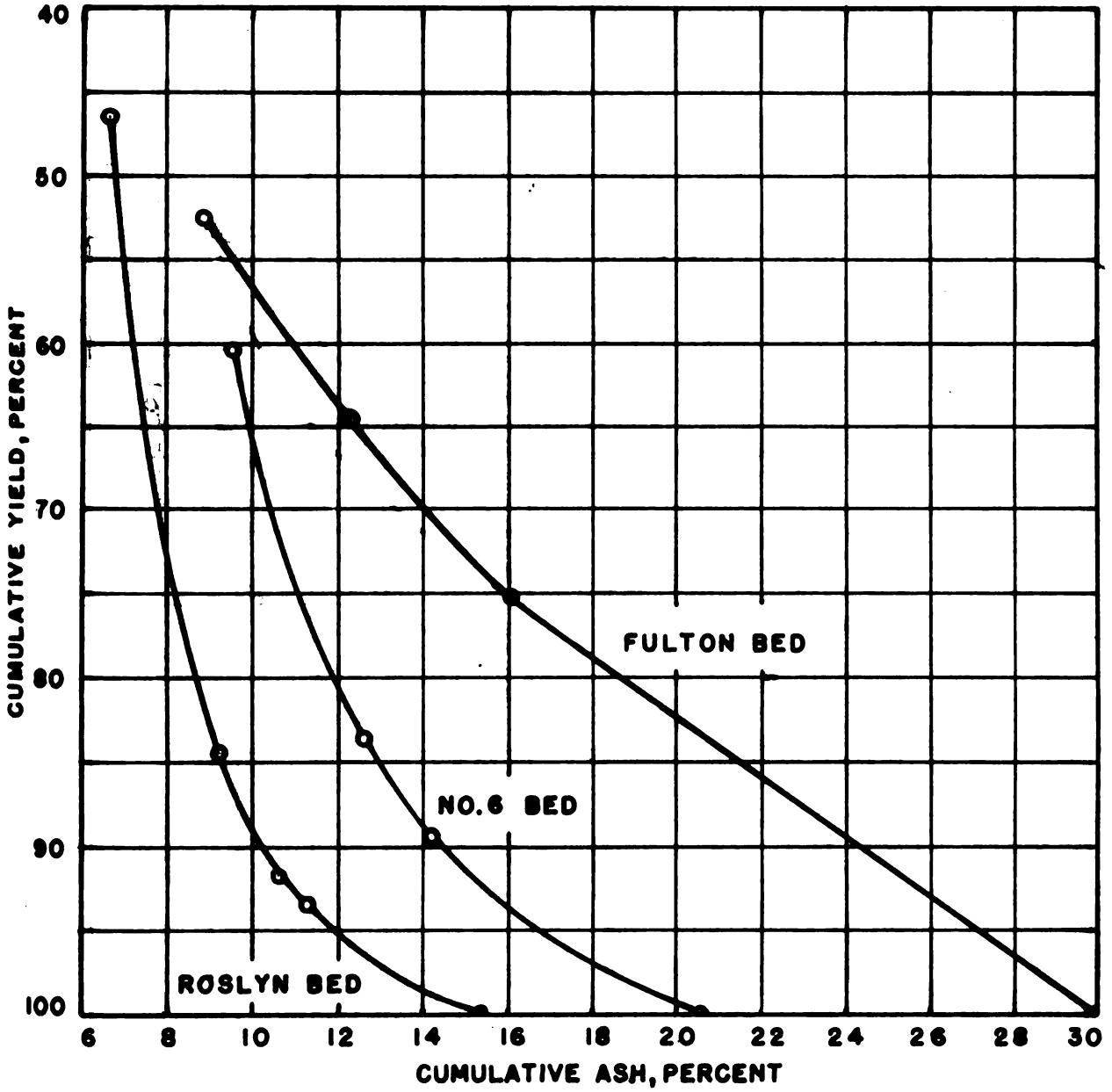

Figure 14. - Float-and-sink yield-ash curves for Roslyn, No. 6, and Fulton beds.

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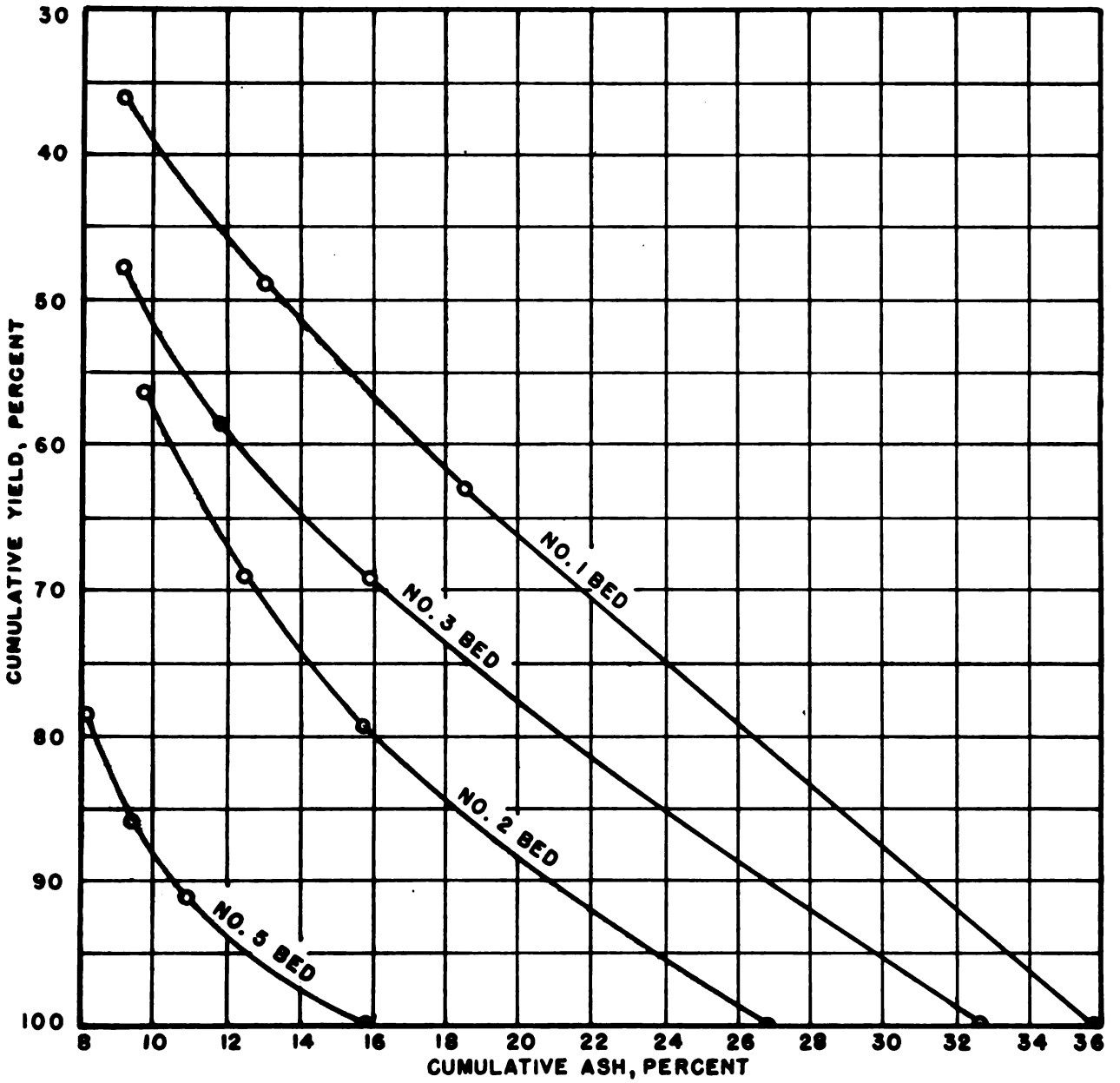

Figure 15. - Float-and-sink yield-ash curves for Wilkeson coal beds.

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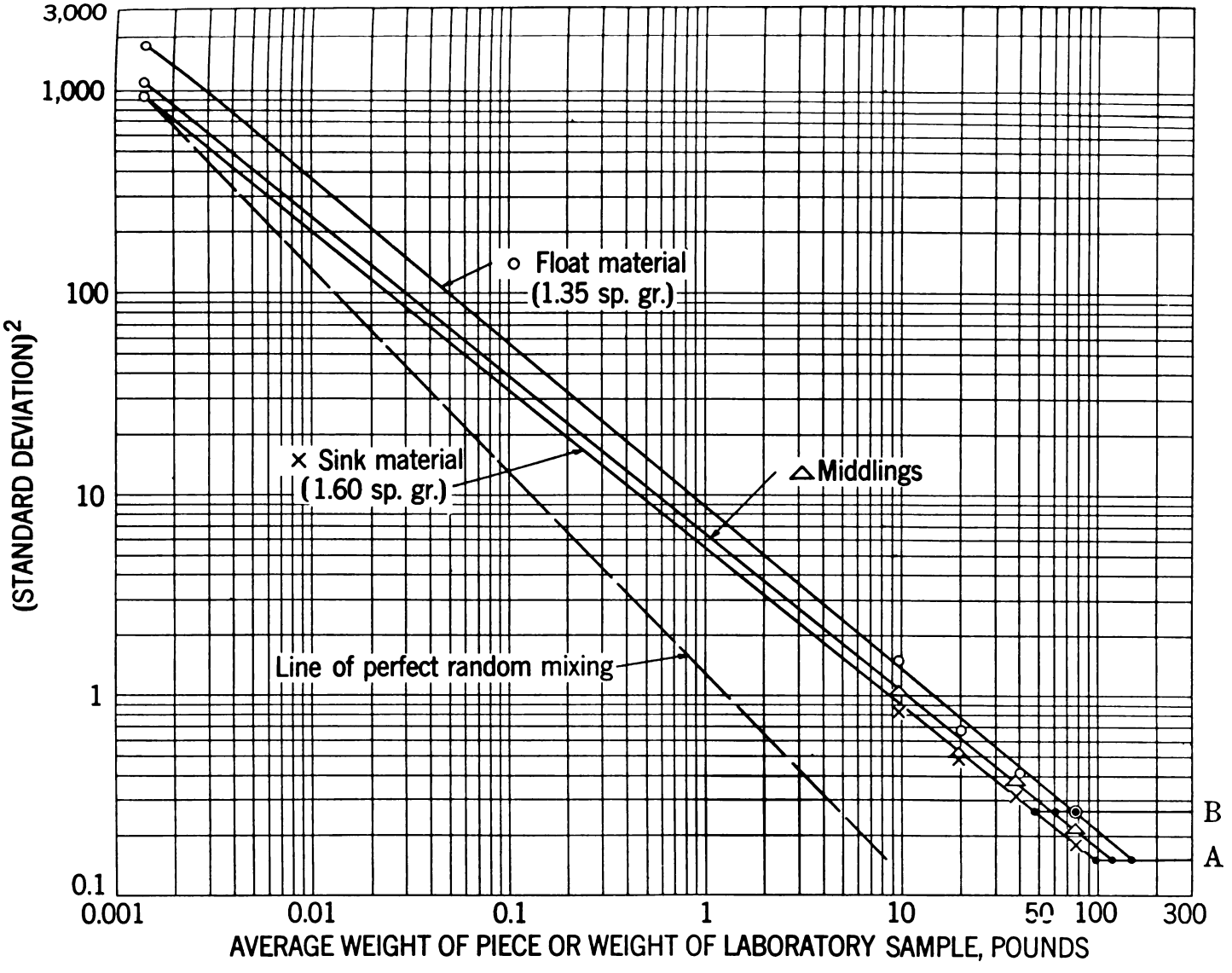

Figure 17. - Sampling characteristics of a western Pennsylvania bituminous coal. Line A refers to an accuracy of ± 1%, 99 times in 100. Line B refers to an accuracy of ± 1%, 95 times in 100.

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TABLE 4. - Specific-gravity analyses of raw coal from Southport mine, Oregon

Size, inches and mesh	Specific gravity	Weight, percent	Ash, 1/ percent	Cumulative Weight, percent	Cumulative Ash, 1/ percent
2 to 1-1/2 inches	Under 1.40	73.2	9.8	73.2	9.8
Weight, 10.9 percent ..	1.40 to 1.50	10.6	22.9	83.8	11.5
Ash, 22.4 percent 1/ ...	1.50 to 1.70	2.8	39.3	86.6	12.4
	Over 1.70	13.4	87.8	100.0	22.4
1-1/2 to 3/4 inch	Under 1.40	72.4	9.8	72.4	9.8
Weight, 21.3 percent ..	1.40 to 1.50	11.2	22.1	83.7	11.4
Ash, 22.1 percent 1/ ...	1.50 to 1.70	3.4	40.3	87.1	12.6
	Over 1.70	13.0	85.9	100.0	22.1
3/4 to 3/8 inches	Under 1.40	72.4	9.4	72.4	9.4
Weight, 23.7 percent ..	1.40 to 1.50	9.9	21.2	85.3	10.8
Ash, 20.1 percent 1/ ...	1.50 to 1.70	3.7	39.0	89.0	11.9
	Over 1.70	11.0	86.3	100.0	20.1
3/8 inch to 20 mesh ...	Under 1.40	72.4	8.2	72.4	8.2
Weight, 36.2 percent ..	1.40 to 1.50	10.1	18.4	82.6	9.4
Ash, 19.3 percent 1/ ...	1.50 to 1.70	3.6	33.6	86.2	10.5
	Over 1.70	13.9	74.0	100.0	19.3
Under 20 mesh	Under 1.40	49.0	8.0	49.0	8.0
Weight, 7.9 percent ...	1.40 to 1.50	11.0	18.4	60.0	9.9
Ash, 34.7 percent 1/ ...	1.50 to 1.70	7.1	32.9	67.1	12.3
	Over 1.70	32.9	80.3	100.0	34.7
Composite, 2 inches to 0	Under 1.40	71.3	9.0	71.3	9.0
Weight, 100.0 percent .	1.40 to 1.50	10.4	20.4	81.7	10.5
Ash, 21.6 percent 1/ ...	1.50 to 1.70	3.8	36.5	85.5	11.6
	Over 1.70	14.5	81.0	100.0	21.7

1/ Moisture-free basis.

Sampling of Coal for Float-and-Sink Tests

Three typical coal samples, one anthracite and two bituminous, representing nationally important deposits, were examined in an intensive experimental study of the variability of several items of analytical data, and the relation of variability to particle size and sample bulk.38/

The data presented covered two of the samples and set up a relationship between particle size and sample size referred to a range of variability tolerance to suit the many situations in which float-and-sink data are used.

The findings are summarized in figures 16 and 17, showing standard deviation from the mean in relation to sample weight for each of the two coals. On these charts, the requirement for a variability tolerance of ± 1 percent of the sample in

38/ Bailey, A. L., and Landry, B. A., Sampling of Coal for Float-and-Sink Tests: Mining Eng., vol. 1, No. 3, 1949; A.I.M.E. Mining Transactions, pp. 79-84; A.I.M.E. T.P. 2539F.

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99 samples out of 100 is shown where the cross line A intersects the curves. Line B similarly indicates the conditions for a precision of ± 1 percent 95 times out of 100.

For the two coals covered by this investigation, the sample weights of sink material indicated for these two precision standards are (1) for the Pittsburgh coal of 1-1/2-inch top size, 96 lb. for 99 out of 100 samples to fall within the tolerance of ± 1 percent sink, and 46 pounds to yield an accuracy of 95 out of 100, and (2) for the anthracite pea size, 130 lb. and 70 lb., respectively, for the above tolerances.

Upgrading Marginal Coking Coals by Coal-Preparation Methods

As part of the study to determine the amount of available coking-coal reserves suitable for the production of metallurgical coke, preparation characteristics of high-sulfur coking coals in southwestern Pennsylvania and northern West Virginia were investigated. It was desired to determine whether the sulfur and ash in these coals can be reduced to meet present metallurgical coking-coal standards by conventional coal-preparation methods and to develop methods that can be used to upgrade high-sulfur and high-ash coals with strong coking properties to make them suitable for metallurgical use.

Production of Electrode Carbon in Germany

Several processes for the production of ultra-clean coal for electrode carbon were developed in Germany prior to and during World War II. These processes have been investigated by British and U. S. Government missions since the end of the war.39/

Almost all the ultra-clean coal was used in the manufacture of electrodes for the production of aluminum and magnesium. Standard raw materials for making these electrodes are pitch coke and petroleum coke, which are nearly ashless. The inclusion of ash in the electrode carbon results in lowered efficiency and impure metal for the reduction process. Petroleum coke and pitch coke were in very short supply in Germany during the war, so that it was necessary to augment the supply with carbon produced from coal. In the emergency, methods were developed to clean coal, which never before had been economically feasible. Other uses of ultra-clean coal are in direct hydrogenation of coal, in production of activated carbon, and for special fuels. Specifications for electrode carbon are rather severe owing to the necessity for producing pure metal.

The most important process for ultra cleaning of coal in Germany was froth flotation. In two plants the coal cleaned by froth flotation was treated with acid or caustic to lower the ash content further. Another process used on an industrial scale was dissolving the coal substance in the middle oil from direct hydrogenation of coal under controlled pressure and temperature. The resulting paste was heated, filtered, and the oil distilled off. The residue was a low-ash organic material that could be carbonized for electrode carbon or could be hydrogenated directly. Several other processes had been developed to the laboratory or pilot-plant stage but had never been used on a commercial scale. These processes included cleaning by centrifugal action, cleaning by electrostatic action, and cleaning by forming a paste with oil in water.

39/ Graham, H. G., and Schmidt, L. D., Methods of Producing Ultra-Clean Coal for Electrode Carbon in Germany: Bureau of Mines Inf. Circ. 7481, 1948, 13 pp.

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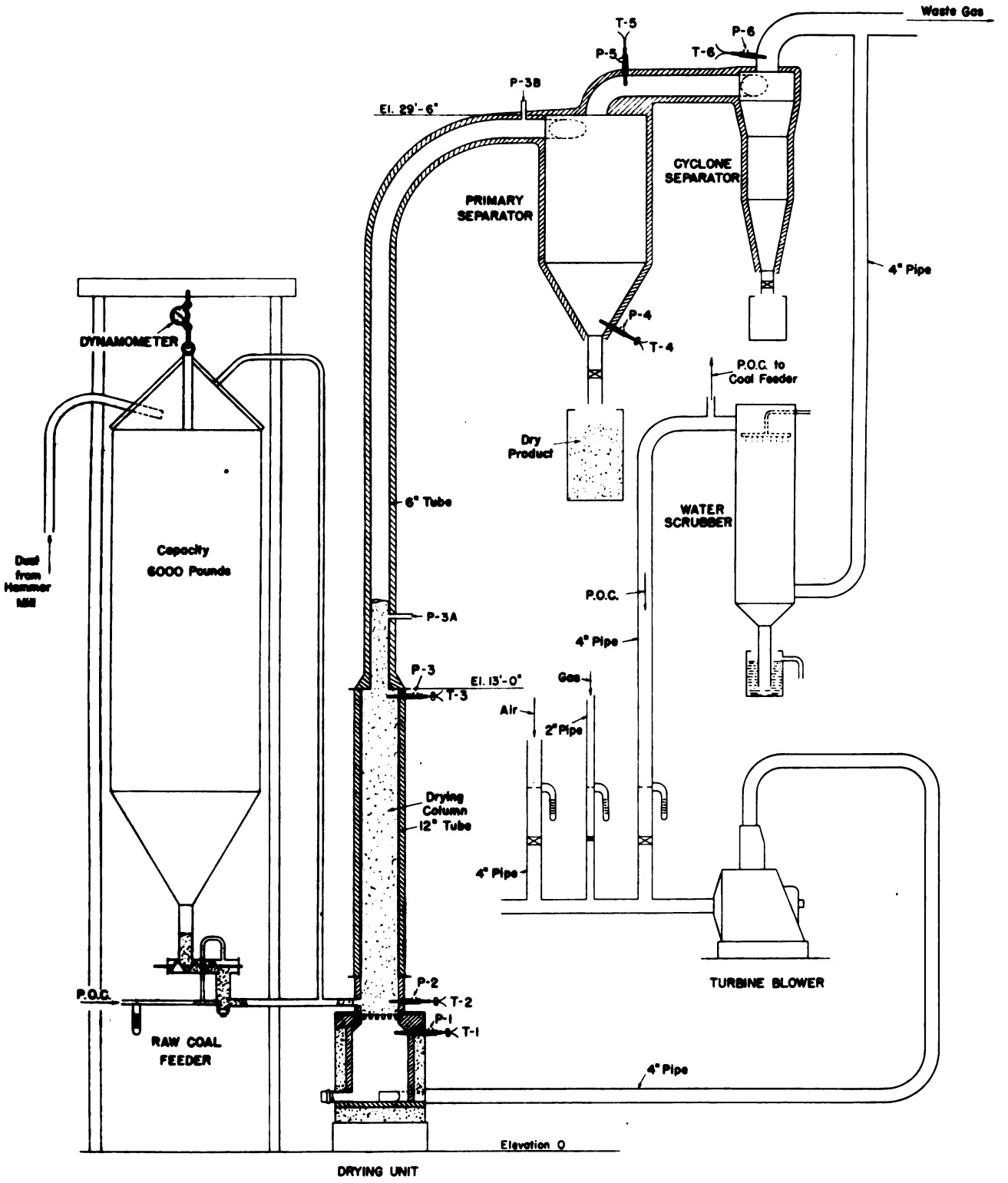

Figure 18. - No. 4 Pilot plant for drying fine coal. Capacity, 2,400 pounds per hour.

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The ultra-clean coal required coking and calcination before it was ready for electrode manufacture. Coking was carried out in conventional-type byproduct ovens at high temperatures. Calcination was effected in continuous-feed, vertical-shaft kilns. When the volatile content of the coke was 5 percent or more, no auxiliary fuel was required to operate the kilns. Coke of less than 5 percent volatile matter required producer gas for heating. The calcined carbon from the kilns was crushed, mixed with pitch, pressed or extended to form electrodes, and baked in resistance-type electric furnaces.

Briquetting of coal

To meet the many requests for information on the briquetting of coal and other carbons, the Bureau of Mines compiled a selected bibliography on the subject.40/

Drying Low-rank Coals

Flash-drying Process

Experimental work on the removal of natural bed moisture from low-rank fine coals was continued with the construction and operation of two additional pilot plants, the later plant having a capacity of about 25 tons per day.

The general problem of drying coals of high bed moisture was discussed.41/ The problem of removing natural moisture is somewhat different than that of removing surface moisture. In the first instance time must be allowed for heat to penetrate into each piece of coal to liberate the moisture, whereas in the latter only sufficient heat must be supplied to evaporate the surface moisture, and the time factor is not so important. Because of the mechanical difficulties involved in handling fine sizes of wet (high-surface-moisture) coal, this study was confined to removal of natural bed moisture of low-rank coals with relatively low surface moisture. A theoretical study of the problem indicated that the time required to heat particles of coal should vary approximately as the square of the diameter of the particle. In other words, a 1/16-inch particle of coal should require about 3 seconds for complete drying at a moderate temperature, whereas a 1/4-inch piece of coal should require about 48 seconds under the same conditions.

A particle of coal can be suspended in a rapidly moving current of hot gas or in a slower moving fluidized bed to achieve drying. The experimental program covered both phases, but during 1949 more attention was devoted to the study of drying in fluidized beds, because there is more latitude in handling larger sizes under these conditions. Experiments made with the pilot plant shown in figure 18 were conducted in fluidized beds on coal sizes up to 1/4 x 0 inch. In this unit, coal is propelled into the drying column pneumatically and comes in contact with jets of high-temperature gases. The coal particles are fluidized and move upward to the cyclone separators. The time of contact of the coal particles and hot gases, ranging from 20 to 50 seconds, is adjusted by controlling the rate of coal and gas feed. In practice, the velocity of gases in the top of the drying column reaches 15 feet per second, which establishes a light-phase fluidized bed, and the time of contact of the particles is adjusted to approach the theoretical time.

40/ Fisher, Paul L., A Selected Bibliography of Briquetting of Coal and Other Carbons: Bureau of Mines Inf. Circ. 7469, 1948, 15 pp.

41/ Parry, V. F., Goodman, J. B., and Wagner, E. O., Drying Low-rank Coals in the Entrained and Fluidized State: Min. Eng., vol. 1, No. 4, 1949, pp. 89-98.

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Experience with the pilot plant has shown that approximately 600 pounds of moisture can be removed from coal per hour per square foot of drying-column area. The unit removes moisture from low-rank coals at efficiencies of about 90 percent, and the process has promise for low-cost drying. Table 4 gives operating data when typical Wyoming subbituminous coal and Texas and Greek lignites were dried in the unit.

Drying and Utilization of Greek Lignites

The Bureau has cooperated with the Economic Cooperation Administration, State Department, in examining Greek fuels to outline methods of production and utilization. Two substantial shipments of low-grade lignite from the Megara and Ptolemais areas of Greece were studied during 1949 to determine how these fuels can be upgraded to make them suitable for industrial purposes. In the natural state, these lignites contain 50 to 62 percent moisture, and it is necessary to dry the lignite before use in the pulverized form.

Drying tests were conducted on the Megara and Ptolemais Greek lignites in the pilot plant. The moisture in the Megara fuel was reduced from 48 to 8.5 percent, and that in the Ptolemais lignite (see table 5) was reduced from 58.8 to 6.8 percent. Several experiments with the drying unit indicated these fuels can be flash-dried readily to low-moisture content to make them suitable for industrial use in pulverized fuel-burning equipment.

A large sample of the dried Megara lignite was pulverized and then burned in a combustion system of 600 cubic foot furnace volume simulating a cement kiln. Flame temperature of 2,700° F. was attained while releasing heat at a rate of about 20,000 B.t.u. per hour per cubic foot. These tests demonstrated that the dried, pulverized Greek lignites are suitable for industrial purposes. Table 5 gives operating data on the drying plant during the processing of the Ptolemais lignite.

When the Ptolemais Greek lignite is dried to 6.8 percent moisture, a fuel of the following proximate analysis results:

	Percent
Moisture	6.8
Volatile matter	44.3
Fixed carbon	33.2
Ash	15.7
	100.0
Sulfur	1.2
B.t.u./lb.	8,760

It is evident that a pulverized fuel of the foregoing analysis will burn satisfactorily in industrial equipment; therefore, no combustion tests were made on the limited sample. After determining that the Ptolemais lignite had a relatively high content of extractable waxes, tests were made to determine the possibilities of briquetting the dried fuel without binder. A substantial sample was briquetted in a high-pressure rotary briquet press to make small briquets for stoker or domestic use. These briquets are satisfactory as regards physical properties if kept covered, but they will disintegrate on exposure to weather. Other small samples of Greek fuels of higher rank than the low-grade lignites were analyzed and tested. It was generally concluded that any of the Greek fuels can be utilized satisfactorily if they are dried before use.

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TABLE 5. - Experimental data on drying subbituminous coal and lignite obtained in Golden, Colo., flash-drying pilot plant No. 4

Kind of coal	Lignite	Lignite	Subbituminous C
Source	Greece	Texas	Wyoming
Materials and moisture data:			
Coal charging rate, lb./hr.	810	1166	1464
Coal charging rate, lb./hr./sq. ft.	1032	1485	1865
Inert gas used for moving coal, cu. ft./lb.	2.6	1.8	1.5
Moisture in raw coal, percent (as charged)	58.8	39.2	30.2
Moisture in dried coal, percent	6.8	3.4	3.2
Dust loss, percent	1.23	0.95	0.21
Heating system data:			
Natural gas used, c.f.h.	749	723	707
Net heat supplied, M B.t.u./hr.	659.1	636.2	622.2
Net heat supplied per lb. of raw coal, B.t.u.	814	546	425
Hot gas used to dry coal, cu. ft./lb.	17.4	12.7	9.8
Products of combustion recirculated, c.f.h.	3813	4561	3938
Mass velocity in column, lb./hr./sq. ft.	1939	1992	1917
Mean space velocity in column, ft./second	14.9	14.2	13.3
Contact time of coal, seconds	19.0	31.6	44.2
Temperature in system, °F.:			
Combustion chamber, point 1	2240	2105	2140
Bottom of column, point 2	990	460	400
Top of column, point 3	410	300	280
Coal from separator, point 4	310	290	270
Gas outlet from separator, point 5	310	290	270
Gas outlet from secondary separator, point 6	310	285	265
Heat balance			
Net heat used, B.t.u./lb. raw coal charged 1/	738	498	380
Net heat required to dry coal, B.t.u./lb. 2/	684	464	355
Drying efficiency, excluding radiation, percent 1/	92.7	93.2	93.4
Over-all efficiency, percent, including radiation	84.0	85.0	83.5

1/ Total net heat, excluding radiation.
2/ Includes heat used for heating coal-carrier gas, excludes radiation.

Drying of Lignite

Several samples of lignite from each of five mines (two underground and three strip mines) from the north, east, central, and southwest regions of the North Dakota deposits, were dried by a modified Fleissner process with steam up to 400 pounds pressure per square inch. Among the drying characteristics quantitatively determined was the steam requirement for two strip-mine lignites, in pounds of steam to remove 1 pound of water. The average figure was found to be 0.8 lb. steam per pound of water removed at pressures between 300 and 400 p.s.i., with very little variation between the two lignites tested.

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STORAGE OF COAL

Storage of Low-Rank Coals

Field investigations on storage of low-grade coals were continued, with periodic observations on two model coal piles, each containing 5,000 tons of subbituminous slack. These remained in excellent condition after a year of storage. The piles were formed under the supervision of Bureau engineers to demonstrate correct methods of storing low-rank coals, and the following general rules were followed in placing the coal:

1. Form the pile in benches of 6 to 12 inches by leveling and compacting with a bulldozer.

2. Distribute each bench of coal to form sides with slope of less than 20°. The sides must be compacted, and it should be feasible to drive a passenger car over any surface of the pile.

3. The leveling and compacting of each bench should be as uniform as possible to minimize segregation and to build up to the maximum density. Proper distribution of the coal should achieve a bulk density of 65 to 70 pounds per cubic foot.

Good progress was made in the storage of lignite at the Garrison Dam in North Dakota. This work is being done in cooperation with the Corps of Engineers, U. S. Army, as part of the Missouri Basin Development Program. During excavation of the dam, approximately 5 million tons of lignite will be uncovered and placed in storage. A demonstration storage pile of lignite comprising about 60,000 tons, formed approximately as outlined above during November 1948, was in good condition on June 30, 1949, but trouble was expected in one section of the pile, where the recommendations for satisfactory storage were not followed. The smaller storage piles of 10,000 to 20,000 tons have been in place for about 18 months with no evidence of spontaneous combustion.

Storage of Dried Lignite

Observation of two storage piles of dried lignite, one continuously exposed to the weather and one protected from the weather, was continued. The exposed pile has been under observation for over 18 months, during which time periodic measurement of both temperature and moisture content have been made. The protected storage pile has been under observation for over a year. During this period of observation there has been no tendency to ignite spontaneously.

During these storage periods there have been fluctuations in the moisture content of both piles, depending on the weather. The moisture content of the exposed pile has fluctuated widely but averages about 17 percent. The moisture content of the protected pile has progressively decreased to an average value of about 11 percent. It has now been established that dried lignite, properly prepared and piled, can be stored successfully.

UTILIZATION OF COAL FOR COMBUSTION

Fuel-Engineering Service

As in previous years, fuel engineering service was rendered to Government establishments in the selection and use of fuel and fuel-burning equipment and in the economical use of steam. Total boiler-plant operating-cost comparisons were made for

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the use of oil, gas, and coal and the type of fuel-burning equipment recommended for 37 projects for the Veterans Administration and 6 for the Department of the Army. Studies of boiler-plant equipment and operations were made at the Lyons, N. J., and Richmond, Va., veterans' hospitals, which resulted in reduction of smoke and recommendations for equipment and fuel purchase that will save $26,000 per year. A steam-production cost survey at the Wildlife Refuge, Carbondale, Ill., requested by the Fish and Wildlife Service, indicated that costs could be reduced $54,000 per year by the installation of suitable steam lines and changing from oil to coal firing. At the request of the Department of the Navy, boiler plant operation and equipment at Naval Barracks, Arlington, Va., were studied, and changes were recommended that will reduce excessive fly-ash emission and fuel costs by $1,600 per year. Acceptance tests were made of steam-generating equipment at the new Federal West-Central Heating Plant, Washington, D. C., at the request of the Federal Works Agency. As a result of these tests, many changes had to be made, which have very greatly improved the performance and reliability of the equipment. Service on 125 special problems was given 34 Government agencies. A representative of the Bureau of Mines served as chairman of the Bureau of Federal Supply interdepartmental Federal Fuel-Purchasing Committee, which made a complete study of past Government coal-purchasing procedures; the work resulted in the establishment of revamped and new procedures, which were adopted throughout the Government service. An information circular[42/] covering basic information on the use of propane and butane fuels was published to satisfy requests from many prospective users. This covered the properties of the fuels, the safety precautions to be observed in their use, the methods of transportation, the various purposes for which the fuels are used, the equipment needed, and the amounts used in the United States. There are also published a bulletin[43/] describing the operations of the National Fuel Efficiency Program during the war, including the functioning of the central office, the division of the Nation into working areas, the acquiring of volunteer workers, the functioning of the area officers, and public-relations work. Representative fuel savings resulting from this program at a number of individual plants are listed, as well as the estimated over-all accomplishments.

Under the Presidential edict of January 17, 1948, "Order for Conservation of Fuel Oil, Gasoline, and Gas," which carried the following clause: "No Federal equipment shall be installed for burning fuel oil or gas, or liquefied petroleum gas, and no permanent building or establishment shall be converted to these fuels, without the prior approval of the Bureau of Mines ...," 425 projects were studied and approvals or disapprovals given. This edict was revoked on January 13, 1949.

Cooperative work with The Air Preheater Corp., of Wellsville, N. Y., on the prevention of deposits and corrosion on air preheaters was continued. Contrary to common belief, extended field trials demonstrated that many special alloy steels now in general use for resisting corrosion rapidly disintegrated in air preheater service. This was particularly true of the high-chromium alloy steels, which failed much more quickly than some of the low-alloy steels. A number of types of coatings on metals also were tried, but these failed rapidly in service.

42/ Barkley, J. F., Questions and Answers on Propane and Butane Fuels: Bureau of Mines Inf. Circ. 7519, 1949, 9 pp.

43/ Barkley, J. F., Cheasley, Thos. C., and Waddell, K. M., The National Fuel Efficiency Program During the War Years 1943-45: Bureau of Mines Bull. 469, 1949, 100 pp.

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Boiler Feed-Water Conditioning

Analyses and resulting recommendations were made on 8,742 samples of boiler water during the fiscal year, as follows: 5,771 from the Army and Air Forces; 1,568 from the Veterans Administration; 264 from the Public Housing Administration; 251 from the Post Office Department; 236 from the District of Columbia; 181 from the Navy Department; 173 from the Office of Indian Affairs; 169 from the Department of Justice; 41 from the Public Health Service; 21 from the Federal Security Agency, 20 from the Department of Agriculture; 19 from the Public Buildings Administration; 16 from the Department of Commerce; 5 from the Bureau of Mines; 3 from the Smithsonian Institution; and 2 from the Legislative Branch. This is about a 7-percent increase over the previous fiscal year.

Reports and recommendations covering 26 analyses of various scales, sludges, and deposits, 8 miscellaneous special analyses, and 11 covering analyses of boiler compounds were made.

Two hundred and sixty-four special Bureau of Mines field water-test kits, 12,005 bottles of chemical reagents (an increase of about 36 percent over the previous fiscal year), and 10,040 test-kit replacement items were distributed to various Government activities.

Visits were made to seven heating plants operated by various Federal agencies in the local area of the District of Columbia to determine difficulties, advise on boiler-water treatment, give instructions in boiler-water testing, and inspect boilers for scale and sludge. At the request of the Veterans Administration, special water surveys were made at its boiler plants at Roanoke, Va., and Sunmount, N. Y., to solve unusual operating difficulties. Consulting service was given to District government on the use of amines and sodium sulfite for boiler-water treatment that saved about $800 per annum; to the Veterans Administration, Fort Howard, Md., on boiler-water treatment and testing that greatly reduced the time for boiler cleaning; to the Public Buildings Administration on tests for phosphate and on return-line corrosion; to the Navy Department on boiler-water testing, on the preparation of a boiler water-treatment manual, on the use of a pH meter, on the construction of color comparators for testing phosphate and tannin in boiler water, and on the treatment of boiler water by passing it through silica spheres; to the Naval Ordnance Plant, Alexandria, Va., on general boiler-water testing; to the Department of the Air Force on the preparation of instructions on the use of sodium silicate to control magnesium phosphate scales; to the Civil Works Department of the U. S. Engineers on general boiler-water treatment and testing; to the Post Office Department on boiler-water treatment for low-pressure boilers, on corrosion of pipe fittings, and on the use of amines for sodium metaphosphate; to the Capitol Power Plant on testing condensate for contamination; to the Korean Power Commission on general boiler-water treatment; to the Canadian Bureau of Mines on the set-up and work of boiler water-conditioning service; to the Government of India on boiler-water testing; to the Children's Hospital, Washington, D. C., on boiler-water testing; to the Boston Navy Yard on boiler-water treatment; to the Public Works Administration on water softening at the Blair House, the present residence of the President; and to the Bureau of Federal Supply on boiler compounds and on feedwater problems at Moffet Field.

Boiler Water Research

Research was continued on the use of amines in boiler water. Corrosion in the piping that returns the condensed steam to the boilers is caused by carbonic acid in the condensate. The acid originates in the carbonate content of the raw make-up water for the boilers; in the boiler, carbonate loses carbon dioxide to the steam,

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and carbonic acid forms when the steam condenses. Chemical treatment neutralizes this acidity; stable, volatile, alkaline chemicals introduced into the boiler with the feed water leave with the steam, and the piping is safeguarded because the condensate is then slightly alkaline. It was found that the more highly volatile amines were especially useful in low-pressure and relatively ventless plants.44/, 45/, 46/

Recommendations resulting from the studies pointed out that cyclohexylamine was especially useful in the lowest pressure group of heating plant boilers, but that plants operated at pressures of 60 p.s.i. and higher probably would find that treatment with morpholine was much less expensive. Tests in three representative Naval heating plants and in three large area-heating systems in housing developments of a midwestern city had shown savings of 30 to 60 percent when morpholine was substituted for cyclohexylamine (Coravol) as the treatment chemical for the prevention of corrosion in condensate return lines. During the studies of chemical treatment, many tests were run to determine the corrosiveness of the condensate. The measured rates of corrosion did not appear to agree too well with the concept that there was a simple relationship between corrosiveness and rate of flow. Also, the condensate temperature seemed to have considerably more influence on corrosion than was indicated by published data. The corrosion studies are being continued under carefully controlled conditions in laboratory-scale apparatus. The amines used for the chemical treatment of steam are relatively expensive, as compared to ammonia. The latter, however, is known to attack brass and other copper alloys under certain conditions of use, and therefore might cause deterioration of valves and other nonferrous components of steam systems. Tests are being made to determine whether there are conditions under which relatively inexpensive ammonia can be used successfully for chemical treatment to prevent corrosion in low-pressure systems.

The invention embodied in U. S. Patent 2,454,258 relates to a method of water treatment to prevent embrittlement cracking (boiler-seam cracking) in boilers operated with distilled water make-up or its equivalent.47/ The basic principle is the elimination of causticity (caustic alkalinity) from the boiler water; instead the alkalinity essential to good boiler operation is provided by trisodium phosphate. Hydrolysis of this phosphate imparts to the water a desirable level of alkalinity, which cannot, however, be increased in the seams to the hydroxide concentration required to produce seam cracking. The patent was assigned to the Government of the United States acting through the Secretary of the Interior and is available for unrestrictive license. The embrittlement-detector-test program correlates the results for tests conducted in over a thousand embrittlement detectors mounted on stationary boilers. These results have shown that the zero-causticity method of boiler-water treatment is very effective for preventing seam cracking. Its usefulness is greatest for boilers operated with distilled water, and there are no operating-pressure limitations. One very useful feature of the method is that no inhibitor is added to the boiler water to increase the total dissolved solids; the use of phosphate instead of caustic alkalinity adds no new chemical to the water, and the total dissolved

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44/ Berk, A. A., and Nigon, J., Amine Volatility and Alkalinity in Relation to Corrosion Control in Steam-Heating Systems: Bureau of Mines Tech. Paper 714, 1948, 63 pp.

45/ Berk, A. A., Corrosion Prevention Through the Chemical Treatment of Steam: Proc. Nat. Dist. Heating Assoc., vol. 38, 1947, pp. 273-292 (issued Sept. 1948).

46/ Berk, A. A., El tratamiento quimico del vapor reduce la corrosion en la caneria de retorno: Ingienieria e Industria, vol. 16, 1948, pp. 79-83.

47/ Schroeder, W. C., and Berk, A. A., Water Treatment to Prevent Embrittlement Cracking: U. S. Patent 2,454,258, Nov. 16, 1948.

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solids in the water are not necessarily increased. The embrittlement detector tests have shown also that sodium nitrate treatment has been entirely effective for preventing seam cracking in boilers operated in a range between 100 and 700 p.s.i. Similar success was obtained with nitrate treatment of locomotive boilers; as a result, a very large reduction in the incidence of seam cracking has been reported by the railroad.48/, 49/ One railroad has reported that the cost of treatment is approximately $14 per locomotive per year, or 20 cents per million gross-ton miles in terms of service.

Smoke Abatement

Smoke-abatement problems continued to hold the interest of various cities that requested Bureau of Mines publications and consulting service. These cities included Winston-Salem, N. C.; Spartanburg, S. C.; Fredericton, New Brunswick; Kansas City, Kans.; Boise, Idaho; Indianapolis and Fort Wayne, Ind.; Utica, N. Y.; Waynesboro, Va.; Moncton, New Brunswick; Charleston, W. Va.; Los Angeles, Calif.; Marietta, Ga.; Gadsden, Ala.; Long Island City, N. Y.; Youngstown, Ohio; Dayton, Ohio, and Washington, D. C. Information and consulting service were also given the County of Allegheny, Pa. In considering smoke problems in the cities of Detroit, Mich., and Windsor, Ontario, Canada, the authorities, having no jurisdiction over vessels in the Detroit River, an international boundary, appealed to the International Joint Commission for help in abating smoke from these vessels, which was polluting the air of these cities appreciably. This Commission, created to handle boundary disputes, is making a study of the matter through a committee of three technical experts from Canada and three from the United States, one of whom is a representative of the Bureau of Mines. The Model Smoke Law Committee of the American Society of Mechanical Engineers, of which a Bureau of Mines engineer is chairman, completed a guide for the use of cities adopting or changing smoke ordinances.50/

Domestic Stoker- and Hand-Firing of Coal; Comparison with Oil

A research program designed to assist householders in selecting automatic coal-burning equipment and to assist coal producers in preparing coal for domestic-stoker use has been completed.51/ Coal of all ranks is available in Washington State. During the study, a total of 165 burning trials, involving the determination of overall efficiencies and all losses, were made on 19 coals mined in Washington and Oregon. The coals ranged in rank from semianthracite through lignite.

The coals were tested at various feed rates, sized and unsized, under various operating conditions, on three stokers, to determine the best methods of burning each kind of coal and the relative suitability of each for the three types of stokers employed. The same hot-water boiler was used in all trials. In addition, seven of the

48/ Berk, A. A., Railroad Boiler Seam Cracking Controlled by Nitrate Treatment: Ind. and Eng. Chem., vol. 40, 1948, pp. 1371-5.

49/ Berk, A. A., Prevention of Seam Cracking in Locomotive Boilers: The Steam Engineer, vol. 18, 1948, pp. 102-104.

50/ Amer. Soc. of Mech. Engs., Example Sections for a Smoke Regulation Ordinance: Inf. Bull., May 1949, 12 pp.

51/ Yancey, H. F., Johnson, K. A., Cordiner, J. B., Jr., Lewis, A. A., and Lunde, K. E., Burning Washington Coals on Different Types of Domestic Stokers in the Same Hot-Water Boiler; Comparison with Hand and Oil Firing: Bureau of Mines Bull. 475, 1949, 96 pp.

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coals were burned in the same boiler by hand firing to obtain a direct comparison with stoker firing, and the same boiler was also used in oil-burning trials of three domestic oil burners. The efficiency of burning coal on the three stokers under favorable conditions was about the same or only slightly lower than with similar good conditions when oil was used as the fuel. Over-all efficiency ranged up to a maximum of about 79 percent with coal and 80 percent with oil.

The seven coals used for the hand-firing trials ranged in rank from medium-volatile bituminous to lignite, and in caking properties from good-caking to noncaking. The caking coals formed coke masses that required considerable attention but delivered heat to the boiler at a relatively uniform rate over a long period. Noncaking coals ignited easily and required little attention during the firing period but tended to burn out more rapidly than caking coals. Virtually no clinkers were formed during any of the tests. The efficiencies were only 3 to 10 percent lower than those for corresponding coals on the clinker-type underfeed stoker, principally because of low firing rates and care in shaking the grates and adding fresh coal. Results of hand-firing and burning on the clinker-type underfeed stoker showed similar relationships between efficiency and rank of coal fired; that is, the efficiency tended to rise as the rank of coal became higher.

When tested in the same boiler as the stokers, three domestic oil burners operated with over-all efficiencies of 78 to 80 percent, about the same as the maximum obtained in favorable medium-rate burning trials on the coal stokers. When the coal stokers were operated intermittently, the losses from combustible and sensible heat in the flue gases remained low, and the over-all efficiencies were only slightly less than those obtained under continuous operation. The fire continued to act as an efficient, positive source of heat throughout the idle periods when fresh coal was not being supplied - that is, when the stoker was not operating - a condition obviously attained only in solid-fuel combustion.

Measurement of the Reactivity of Solid Fuels by the Crossing-Point Method

The reactivity of coal is one of the properties considered in the evaluation of the suitability of given coals for particular commercial applications, and the need has long existed for a convenient, rational method for determining this property of solid fuels. Many different procedures have been developed and recommended for the purpose of determining the reactivity of coals, but some serious objections to all of them have been raised. The crossing-point method seemed to be most promising, and this method was investigated with the objective of developing it into an acceptable procedure.

The experimental factors affecting the crossing-point test were studied systematically, and several deficiencies of the original procedure were disclosed. On the basis of this study, modification of the procedure was devised.52/ Rigorous examination showed that this method had the necessary qualification of reproducibility and consistency for a standard method. By eliminating much of the error of temperature measurement of the old procedure, the new procedure reduces to an acceptable minimum the effects of unavoidable differences in apparatus construction and permits absolute measurements to be realized.

52/ Jonakin, J., Cohen, P., Corey, R. C., and Jain, B. C., Measurement of the Reactivity of Solid Fuels by the Crossing-Point Method: Proc. Am. Soc. Test. Mat., vol. 48, 1948, pp. 1269-1289.

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The Bureau of Mines, cooperating with the ASME Special Research Committee on Furnace-Performance Factors, began the first phase of an investigation of such factors. Improved methods of measuring temperature and composition of furnace gases were applied to the determination of heat absorption in industrial furnaces. The study of one unit, a pulverized-coal-fired boiler using tangential burners, has been completed 53/ and work on a second unit is in progress.

Corrosion of High-Pressure Steam Generators

Numerous cases of severe, pit-type, internal corrosion in high-pressure steam generators, principally of furnace-wall tubes, have been a source of concern to operators of high-capacity steam boilers. This corrosion has been ascribed to deposits consisting almost entirely of Fe_3O_4 and copper and its oxides. Opinions of the cause of this attack vary widely, but in the absence of conclusive experimental proof that thin deposits of Fe_3O_4, copper, or mixtures of these substances will cause pitting of steel under boiler conditions, the basis of the theories that have been proposed necessarily have been speculative. Published discussions of this problem have been reviewed critically, 54/ and an attempt has been made to correlate the experience with this type of corrosion and the meager amount of available theoretical data. This was to provide an objective background to the problem and to evoke discussion from others who may have the evidence that is needed to establish definitely the effect of deposits of Fe_3O_4 and copper in boilers.

CARBONIZATION OF COAL

Survey of Carbonizing Properties of American Coals

The scope of the survey of the carbonizing properties of American coals was narrowed at the start of the past fiscal year by limiting experimental carbonization to tests at high temperatures. Emphasis was placed upon research bearing directly on the manufacture of metallurgical coke. Coals of low-, medium-, and high-volatile bituminous rank were carbonized, both singly and as blends with coals of different rank. A relatively high proportion of the tests were made on blends, because most metallurgical coke is made from mixed coals; furthermore, the carbonizing properties of some coals carbonized singly are of little value in predicting the physical properties of coke made from their blends.

The source of coals and composition of blends investigated during the past year are given in table 6. Proximate and ultimate analyses of the coals are given in table 7.

The coke-, gas-, and byproduct-making properties of 20 coals were determined. Six were low-volatile coals, all from West Virginia, representing the following beds: Davy Sewell, Pocahontas No. 3, and Pocahontas No. 4, McDowell County; and Pocahontas No. 6 (three samples), Wyoming and Mercer Counties. Four were medium-volatile coals

53/ Reid, W. T., Cohen, P., and Corey, R. C., An Investigation of the Variation in Heat Absorption in a Pulverized-Coal-Fired, Water-Cooled Steam-Boiler Furnace. Part II. Furnace Heat Absorption Efficiency, as Shown by the Temperature, Composition, and Flow of Gases Leaving the Furnace: Trans. A.S.M.E., vol. 70, No. 5, 1948, pp. 569-585.

54/ Corey, R. C., Corrosion of High-Pressure Steam Generators; Status of Our Knowledge of the Effect of Copper and Iron Oxide Deposits in Steam-Generating Tubes: Proc. Am. Soc. Test. Mat., vol. 48, 1948, pp. 907-941.

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representing the Mary Lee bed, Jefferson County, Ala.; Lower Kittanning and Upper Kittanning beds, Clearfield County, Pa.; and Fire Creek bed, Greenbrier County, W. Va. Ten coals of high-volatile rank included the Upper and Lower Freeport beds, Indiana County, Pa.; Pittsburgh bed, Fayette County, Pa.; Imboden bed, Letcher County, Ky.; No. 5 bed, Saline County, Ill.; Eagle bed, Fayette County, W. Va.; No. 2 Gas bed, Kanawha County, W. Va.; No. 5 Block bed, Wayne County, W. Va.; Stockton-Lewiston bed, Lincoln County, W. Va.; and an unnamed bed sampled by core-drilling in Gunnison County, Colo. Other Eagle and Pocahontas No. 3 coals constituted a byproduct-plant blend that was mixed at the plant. Sixteen of these coals were also carbonized as blends with coals of different rank.

TABLE 6. - Description of coals and blends

Coal No.	Description
	Low-volatile coals
345	Davy Sewell bed, Twin Branch mine, McDowell County, W. Va.
345A	Blend: 80 percent Pittsburgh (28) and 20 percent Davy Sewell (345).
347	Pocahontas No. 6 bed, Black Eagle No. 1 mine, Wyoming County, W. Va.
347A	Blend: 80 percent Pittsburgh (28) and 20 percent Pocahontas No. 6 (347).
357	Pocahontas No. 3 bed, Lake Superior No. 3 mine, McDowell County, W. Va., 1.5 specific gravity float.
a357 b357 c357	Pocahontas No. 3 bed. Special mine samples.
358	Pocahontas No. 4 bed, Lake Superior No. 4 mine, McDowell County, W. Va., 1.5 specific-gravity float.
a358 b358 c358	Pocahontas No. 4 bed. Special mine samples.
369	Pocahontas No. 6 bed, Black Eagle No. 1 mine, Wyoming County, W. Va.
369A	Blend: 80 percent Pittsburgh (28) and 20 percent Pocahontas No. 6 (369).
370	Pocahontas No. 6 bed, Louisville mine, Mercer County, W. Va.
370A	Blend: 80 percent Pittsburgh (28) and 20 percent Pocahontas No. 6 (370).
372	Lower Kittanning (B or Miller) bed, Eureka No. 40 mine, Somerset County, Pa.
	Medium-volatile coals
353	Mary Lee bed, Jefferson County, Ala. Blend: 65 percent Sayerton mine and 35 percent Sayre mine.
374	Lower Kittanning (B or Miller) bed, Springfield No. 4 mine, Clearfield County, Pa.
374A	Blend: 80 percent Pittsburgh (28) and 20 percent Lower Kittanning (374).
374B	Blend: 70 percent Pittsburgh (28) and 30 percent Lower Kittanning (374).
375	Upper Kittanning (C) bed, Springfield No. 6 mine, Clearfield County, Pa.
375A	Blend: 80 percent Pittsburgh (28) and 20 percent Upper Kittanning (375).
375B	Blend: 70 percent Pittsburgh (28) and 30 percent Upper Kittanning (375).
378	Fire Creek bed, Laurel Creek mine, Greenbrier County, W. Va.
378A	Blend: 80 percent Pittsburgh (28) and 20 percent Fire Creek (378).

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TABLE 6. - Description of coals and blends (Cont'd.)

Coal No.	Description
	Medium-volatile coals (Cont'd.)
378B	Blend: 70 percent Pittsburgh (28) and 30 percent Fire Creek (378).
	High-volatile coals
b326	Upper and Lower Freeport beds, Kent Nos. 1 and 2 mines, Indiana County, Pa.
a331	Upper Freeport bed, Watson mine, Indiana County, Pa.
346	Upper and Lower Freeport bed, Kent Nos. 1 and 2 mines, Indiana County, Pa.
346A	Blend: 30 percent Upper and Lower Freeport (346) and 70 percent Pittsburgh (28).
346B	Blend: 70 percent Upper and Lower Freeport (346) and 30 percent Pittsburgh (28).
348	Pittsburgh bed, Banning mine, Fayette County, Pa.
352	Core-drill hole No. 26-23, Project 821, Lower bed, Gunnison County, Colo.
356	Imboden bed, Shady Side mine, Letcher County, Ky.
356A	Blend: 80 percent Imboden (356) and 20 percent Pocahontas No. 3 (75).
356B	Blend: 70 percent Imboden (356) and 30 percent Pocahontas No. 3 (75).
361	Illinois No. 5 bed, Sahara No. 16 mine, Saline County, Ill.
361A	Blend: 80 percent Illinois No. 5 (361) and 20 percent Pocahontas No. 3 (75).
361B	Blend: 70 percent Illinois No. 5 (361) and 30 percent Pocahontas No. 3 (75).
362	Eagle bed, Cannelton No. 3 mine, Fayette County, W. Va.
362A	Blend: 40 percent Eagle (362), 40 percent No. 2 Gas (363), 10 percent Pocahontas No. 3 (357), and 10 percent Pocahontas No. 4 (358).
362B	Blend: 35 percent Eagle (362), 35 percent No. 2 Gas (363), 15 percent Pocahontas No. 3 (357), and 15 percent Pocahontas No. 4 (358).
362C	Blend: 40 percent Eagle (362), 40 percent No. 2 Gas (363), and 20 percent Pocahontas No. 3 (75).
362D	Blend: 80 percent Eagle (362) and 20 percent Pocahontas No. 3 (357).
362E	Blend: 35 percent Eagle (362), 35 percent No. 2 Gas (363), and 30 percent Pocahontas No. 3 (75).
362F	Blend: 80 percent Eagle (362) and 20 percent Pocahontas No. 4 (358).
363	No. 2 Gas bed, Cannelton No. 100 mine, Kanawha County, W. Va.
363A	Blend: 80 percent No. 2 Gas (363) and 20 percent Pocahontas No. 3 (357).
364	No. 5 Block bed, Northland mine, Wayne County, W. Va.
366	Stockton-Lewiston bed, H. G. Jones mine, Lincoln County, W. Va.
371	Thick Freeport bed, Harmar mine "low coal," Allegheny County, Pa.
371A	Blend: 82.5 percent Thick Freeport (371) and 17.5 percent Lower Kittanning (372).
a371 b371 c371 d371 e371 f371	Thick Freeport bed, Harmar mine, Allegheny County, Pa. Special channel samples.

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TABLE 6. - Description of coals and blends (Cont'd.)

Coal No.	Description
	High-volatile coals (Cont'd.)
a371A	Blend: 82.5 percent Thick Freeport (a371) and 17.5 percent Lower Kittanning (372).
b371A	Blend: 82.5 percent Thick Freeport (b371) and 17.5 percent Lower Kittanning (372).
c371A	Blend: 82.5 percent Thick Freeport (c371) and 17.5 percent Lower Kittanning (372).
d371A	Blend: 82.5 percent Thick Freeport (d371) and 17.5 percent Lower Kittanning (372).
e371A	Blend: 82.5 percent Thick Freeport (e371) and 17.5 percent Lower Kittanning (372).
f371A	Blend: 82.5 percent Thick Freeport (f371) and 17.5 percent Lower Kittanning (372).
373	Blend: 82.5 percent Thick Freeport, Harmar Mine (full seam) and 17.5 percent Lower Kittanning (372).
376	Upper Freeport bed, Sigley mine, Preston County, W. Va.
	Miscellaneous
354	J. & L. Blend: 80 percent Eagle beds and 20 percent Pocahontas No. 3.
	Standard blending coals
28	Pittsburgh bed, Warden mine, Allegheny County, Pa.
75	Pocahontas No. 3 bed, Kimball, McDowell County, W. Va.

The expanding properties of all coals and blends carbonized by the BM-AGA method (except those tested under cooperative agreement and the drilled sample) were determined. In addition to those listed in the preceding paragraph, expansion tests were made on three special samples of the Pocahontas No. 3 and No. 4 beds and on samples representing the following beds: Upper Freeport, Indiana County, Pa.; Upper Freeport, Preston County, Pa.; Thick Freeport, Allegheny County, Pa. (five samples); and Lower Kittanning, Somerset County, Pa. Eight blends of Thick Freeport and Lower Kittanning coals also were tested.

Methods of Testing

In the survey of reserves of coking coals, the experimental procedure followed heretofore[55/] was modified as follows: (1) BM-AGA tests were made only in 18-inch standard retorts, (2) coals and blends were carbonized at 800° and 900° C., excepting a few samples that were too small, and (3) physical properties (table 8) of the cokes were determined by standard methods. Only three coals and one blend were carbonized in the 500-pound slot oven, for it has been shown that the physical properties of the coke from this oven are intermediate between those of 800° and 900° C. BM-AGA cokes. Expanding properties were determined largely by tests in the sole-heated oven; three coals and one blend were tested in the slot-type expansion oven. The plastic properties of all the separate coals and blends were determined by the Gieseler and Davis methods.

55/ Reynolds, D. A., and Holmes, C. R., Procedure and Apparatus for Determining Carbonizing Properties of American Coals by the Bureau of Mines - American Gas Association Method: Bureau of Mines Tech. Paper 685, 1946, 35 pp.

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TABLE 7. - Analyses of coals, as-carbonized basis[1/]

Coal No.	Dry, mineral matter-free, fixed carbon percent	Proximate, percent			Ultimate, percent						Air drying loss, percent	Heating value, B.t.u. per lb.	Softening temperature of ash, °F.
		Moisture	Volatile matter	Fixed carbon	Ash	Hydrogen	Carbon	Nitrogen	Oxygen	Sulfur			
Low-volatile coals													
345	79.8	1.7	19.1	72.2	7.0	4.7	82.5	1.4	3.5	0.9	0.9	14,290	2,620
347	81.1	1.4	17.9	73.4	7.3	4.3	82.8	1.3	3.5	.8	.7	14,310	2,680
357	84.0	1.1	15.5	78.3	5.1	4.3	85.1	1.2	3.6	.7	.6	14,650	2,420
a357	83.8	1.5	15.7	78.6	4.2	4.4	85.7	1.1	4.0	.6	1.0	14,820	-
b357	83.6	1.4	15.9	78.3	4.4	4.3	85.7	1.1	3.9	.6	.8	14,750	-
c357	84.2	1.8	15.2	78.2	4.8	4.4	85.1	1.1	3.8	.8	1.1	14,640	-
358	81.2	1.0	18.1	75.8	5.1	4.5	84.8	1.2	3.7	.7	.6	14,780	2,450
a358	82.3	1.4	17.1	77.3	4.2	4.5	85.5	1.1	4.1	.6	.8	14,830	-
b358	81.4	1.5	17.9	76.3	4.3	4.5	85.1	1.2	4.3	.6	.8	14,760	-
c358	81.6	1.3	17.7	75.9	5.1	4.5	84.3	1.2	4.3	.6	.7	14,630	-
369	81.4	4.1	17.0	69.9	9.0	4.5	78.4	1.2	6.1	.8	3.7	13,660	2,650
370	80.7	4.4	17.9	72.1	5.6	4.8	81.7	1.2	6.1	.6	3.9	14,220	2,540
372	82.5	2.3	16.5	73.1	8.1	4.5	80.7	1.3	4.0	1.4	1.9	14,010	-
Medium-volatile coals													
353	69.2	6.1	26.5	57.1	10.3	-	-	-	-	.8	5.2	12,840	2,730
374	76.8	1.7	21.9	69.7	6.7	4.8	81.6	1.3	3.8	1.8	1.3	14,430	2,340
375	73.9	1.1	24.8	68.1	6.0	5.0	82.3	1.5	4.3	.9	.6	14,510	2,780
378	76.9	2.8	21.7	70.3	5.2	4.9	82.7	1.4	5.0	.8	2.1	14,430	2,910
High-volatile coals													
b326	67.8	3.4	29.1	59.4	8.1	5.1	77.0	1.4	7.0	1.4	2.5	-	-
a331	67.8	3.6	29.0	59.2	8.2	5.2	76.5	1.4	7.8	.9	2.5	-	-
346	66.4	1.7	31.2	60.0	7.1	5.2	79.3	1.4	5.1	1.9	.8	14,110	2,450
348	64.0	2.6	32.8	56.6	8.0	5.2	76.0	1.6	8.2	1.0	1.3	13,580	2,650
352	57.6	2.2	39.5	52.8	5.5	5.6	76.6	1.7	10.0	.6	.7	13,730	2,420
356	61.8	3.0	35.8	57.0	4.2	5.6	78.0	1.6	9.6	1.0	1.3	13,910	2,840
361	60.9	6.4	34.4	52.3	6.9	5.4	71.2	1.6	13.2	1.7	3.3	12,680	2,160

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TABLE 7. - Analyses of coals, as-carbonized basis[1] (Cont'd.)

Coal No.	Dry, mineral matter-free, fixed carbon percent	Proximate, percent			Ultimate, percent						Air drying loss, percent	Heating value, B.t.u. per lb.	Softening temperature of ash °F.
		Moisture	Volatile matter	Fixed carbon	Ash	Hydrogen	Carbon	Nitrogen	Oxygen	Sulfur			
High-volatile coals - (Cont'd.)													
362	63.7	1.9	34.1	58.7	5.3	5.4	79.4	1.5	7.7	0.7	0.6	14,130	2,910
363	61.0	1.7	36.6	56.3	5.4	6.0	77.4	1.5	8.6	1.1	.8	14,070	2,780
364	59.5	4.9	34.8	49.2	11.1	5.1	69.4	1.3	11.7	1.4	1.2	12,270	2,730
366	57.7	4.4	36.4	47.7	11.5	5.2	68.3	1.3	11.6	2.1	1.7	12,320	2,700
371	62.4	2.4	32.4	51.1	14.1	4.9	70.0	1.3	7.9	1.8	1.0	-	-
b371	62.0	2.6	35.0	55.9	6.5	5.4	77.0	1.5	8.2	1.4	.9	-	-
c371	61.8	2.5	35.4	56.2	5.9	5.5	77.4	1.5	8.4	1.3	.9	-	-
d371	61.5	3.4	35.1	54.7	6.8	5.5	75.5	1.5	9.0	1.7	2.0	13,610	-
e371	62.0	4.1	33.6	53.1	9.2	5.3	73.2	1.4	9.4	1.5	2.9	13,170	-
f371	63.0	5.3	33.1	55.1	6.5	5.6	74.9	1.5	10.5	1.0	4.1	13,420	-
373	65.6	2.4	30.9	56.8	9.9	5.0	75.1	1.4	7.3	1.3	1.3	-	-
376	68.2	.8	28.2	57.3	13.7	4.7	73.8	1.3	5.2	1.3	.0	13,160	-
Miscellaneous													
354	69.1	3.5	27.5	59.3	9.7	-	-	-	-	0.7	2.3	13,300	2,570
Standard blending coals													
28	62.8	1.3	34.9	57.8	6.0	5.3	79.3	1.6	7.0	0.8	0.4	14,090	2,800
75	82.3	2.3	16.7	74.5	6.5	4.4	82.5	1.6	4.4	.6	1.8	14,240	2,350

1/ Analyses made under supervision of H. M. Cooper, chemist, Bureau of Mines.

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TABLE 8. - Physical properties of coke (A. S. T. M. standard method)

Coal No.	Carbonizing temperature, °C.	True specific gravity	Apparent specific gravity	Cells, percent	Shatter test, cumulative percent upon - 2-inch screen	1-1/2-inch screen	1-inch screen	1/2-inch screen	Tumbler test, cumulative percent upon - 2-inch screen	1-1/2-inch screen	1-inch screen	1/2-inch screen	1/4-inch screen
Low-volatile coals													
345A	800	1.85	0.85	54.1	64	85	91	92	6	29	56	63	64
345A	900	1.87	.86	54.0	50	82	95	98	0	9	49	64	66
347A	800	1.88	.85	54.8	81	94	97	98	9	38	56	61	61
347A	900	1.90	.88	53.7	53	82	95	98	5	16	52	64	65
357	800	1.83	.89	51.4	85	92	94	95	7	39	49	50	50
357	900	1.88	.90	52.1	57	83	96	97	1	16	53	61	62
358	800	1.84	.85	53.8	84	95	98	99	17	47	59	61	61
358	900	1.89	.86	54.5	58	87	97	99	1	20	56	65	66
369A	900	1.89	.83	56.1	54	86	96	98	1	22	54	63	65
370A	800	1.85	.82	55.7	86	94	97	98	15	44	57	61	61
370A	900	1.89	.82	56.6	56	84	96	98	1	17	53	62	64
Medium-volatile coals													
353 [1]	800	1.89	0.77	59.3	85	97	98	98	26	45	54	56	56
353 [2]	800	-	.91	-	84	95	98	98	13	48	58	62	62
374	900	1.90	.83	56.3	61	89	96	98	3	32	56	62	63
374A	800	1.86	.86	53.8	76	90	96	98	4	29	52	62-	63
374A	900	1.88	.87	53.7	39	76	94	98	-	9	48	64	65
374B	800	1.86	.85	54.3	80	91	96	98	9	31	56	63	64
374B	900	1.87	.86	54.0	49	83	95	98	1	16	52	65	67
375	900	1.89	.82	56.6	65	87	97	98	6	27	57	63	64
375A	800	1.87	.87	53.5	72	89	95	97	4	24	48	61	63
375A	900	1.87	.88	52.9	37	77	96	98	-	11	50	63	65
375B	800	1.87	.84	55.1	78	91	96	98	7	30	52	62	63
375B	900	1.87	.87	53.5	43	77	96	98	-	13	50	64	65
378	900	1.89	.85	55.0	64	88	96	98	2	21	51	59	60
378A	800	1.86	.86	53.8	74	90	96	98	3	27	54	63	64
378A	900	1.88	.87	53.7	45	79	95	98	-	10	50	64	66
378B	900	1.88	.85	54.8	51	84	96	98	1	16	52	64	65

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TABLE 8. - Physical properties of coke (A. S. T. M. standard method) (Cont'd.)

Coal No.	Carbonizing temperature, °C.	True specific gravity	Apparent specific gravity	Cells, percent	Shatter test, cumulative percent upon -				Tumbler test, cumulative percent upon -				
					2-inch screen	1-1/2-inch screen	1-inch screen	1/2-inch screen	2-inch screen	1-1/2-inch screen	1-inch screen	1/2-inch screen	1/4-inch screen
					High-volatile coals								
346 [3]	900	1.89	0.84	55.6	56	85	95	-	2	21	52	61	63
346A [3]	900	1.87	.86	54.0	48	77	94	-	0	11	39	57	59
346B [3]	900	1.89	.84	55.6	52	81	95	-	0	14	47	58	61
348 [3]	800	1.88	.90	52.1	62	84	93	-	4	35	56	-	67
348 [3]	900	1.92	.88	54.2	43	76	93	-	1	23	55	-	68
348 [3]	1000	1.92	.85	55.7	14	56	87	-	0	7	44	-	69
352 [4]	900	1.91	.74	61.3	-	23	72	94	-	6	53	81	-
356	800	1.84	.77	58.2	56	76	89	96	-	8	24	53	62
356	900	1.84	.78	57.6	33	68	91	97	-	3	34	63	67
356 [5]	870-1,010	1.86	.80	57.0	51	76	91	97	-	5	25	56	62
356A	800	1.87	.81	55.5	79	90	96	97	3	28	48	58	60
356A	900	1.87	.80	57.2	43	77	95	98	-	9	45	63	65
361	800	1.87	.77	58.8	51	73	89	97	0	4	22	56	66
361	900	1.91	.78	59.2	31	65	91	97	-	3	31	63	68
361 [5]	870-1,010	1.90	.80	57.9	42	71	91	97	1	5	29	61	68
361A	800	1.87	.81	56.7	83	93	97	98	10	36	55	60	61
361A	900	1.90	.77	59.5	45	79	95	98	-	11	50	63	65
361A [5]	870-1,010	1.90	.79	58.4	69	89	98	100	7	21	52	59	61
362	800	1.84	.78	57.6	73	87	94	97	3	20	42	53	61
362	900	1.86	.82	55.9	43	77	94	97	0	9	52	66	68
362A	800	1.84	.82	55.4	84	95	97	98	14	46	60	63	63
362A	900	1.89	.83	56.1	42	83	96	98	0	16	56	67	68
362B	800	1.86	.84	54.8	82	95	98	98	12	46	61	64	64
362B	900	1.88	.86	54.3	42	81	96	98	1	16	55	68	69
362C	800	1.86	.84	54.8	83	95	97	98	9	44	59	62	62
362C	900	1.88	.83	55.9	47	81	96	98	0	13	52	66	67
362D	800	1.86	.82	55.9	83	94	97	98	6	43	61	65	65
362D	900	1.89	.83	56.1	44	79	96	98	1	13	55	67	69
362F	800	1.86	.81	56.5	84	93	93	94	8	42	61	63	64
362F	900	1.89	.80	57.7	38	78	96	98	-	14	54	67	69
363	800	1.86	.83	55.4	68	84	92	96	1	12	34	54	59

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TABLE 8. - Physical properties of coke (A. S. T. M. standard method) (Cont'd.)

Coal No.	Carbonizing temperature, °C.	True specific gravity	Apparent specific gravity	Cells, percent	Shatter test, cumulative percent upon - 2-inch screen	Shatter test, cumulative percent upon - 1-1/2-inch screen	Shatter test, cumulative percent upon - 1-inch screen	Shatter test, cumulative percent upon - 1/2-inch screen	Tumbler test, cumulative percent upon - 2-inch screen	Tumbler test, cumulative percent upon - 1-1/2-inch screen	Tumbler test, cumulative percent upon - 1-inch screen	Tumbler test, cumulative percent upon - 1/2-inch screen	Tumbler test, cumulative percent upon - 1/4-inch screen
					High-volatile coals (Cont'd.)								
363	900	1.86	0.86	53.8	36	72	93	97	0	3	31	54	58
363 5/	870-1,010	1.88	.87	53.7	59	83	94	97	0	10	38	55	59
363A	900	1.86	.84	54.8	44	80	96	98	1	15	53	65	67
364	800	1.89	.83	56.1	65	80	87	91	-	5	19	39	49
364	900	1.94	.82	57.7	58	81	91	95	-	4	24	44	52
364	1,000	1.90	.83	56.3	37	68	90	96	-	3	27	52	58
366	800	1.90	.77	59.5	76	84	90	93	2	9	23	41	48
366	900	1.96	.82	58.2	65	84	92	95	-	9	34	48	53
366	1,000	1.89	.82	56.6	42	75	92	96	-	5	33	55	59

1/ Moisture in coal, 6.1 percent.
2/ Moisture in coal, 1.4 percent.
3/ Cokes tested by Bureau of Mines method.
4/ Cokes tested by Columbia Steel Co. method.
5/ Carbonized in 500-pound slot oven.

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In analyzing the results of laboratory carbonization tests, the physical properties of the cokes and expansion of the coals are stressed, because these factors are of prime importance in commercial plants. Yields, which are of secondary importance, are given in table 9.

TABLE 9. - Yields of carbonization products, as-carbonized basis

Coal No.	Carbonizing temperature, °C.	Coke, percent	Yields per ton of coal 1/				
			Gas, cubic feet	Tar, gallons	Light oil, gal.		$(NH_4)_2SO_4$, pounds
					In gas	Tar to 170° C.	
Low-volatile coals							
345A	800	72.3	8,700	14.5	2.00	0.84	27.1
345A	900	71.7	10,100	-	2.61	-	24.1
347A	800	72.3	9,100	15.0	1.83	.81	26.2
347A	900	71.9	10,400	12.6	2.78	.47	23.5
357	800	83.9	8,950	3.1	1.02	.09	12.6
357	900	84.1	10,450	2.8	1.14	.04	7.8
358	800	83.1	9,100	5.1	1.29	.23	17.4
358	900	82.6	10,200	5.2	1.36	.12	12.1
369A	900	71.9	10,000	12.4	2.43	.82	23.5
370A	800	71.7	8,850	15.4	2.28	1.16	25.6
370A	900	71.5	10,100	12.9	2.58	.81	22.5
Medium-volatile coals							
353 2/	800	71.5	8,550	11.9	-	0.50	29.8
353 3/	800	75.0	9,100	12.0	-	.49	29.2
374	900	78.1	10,400	8.4	1.97	.29	17.1
374A	800	71.2	9,350	15.3	2.44	1.16	26.6
374A	900	71.1	10,550	13.4	2.98	.70	23.9
374B	800	72.6	9,000	15.1	2.67	1.09	23.8
374B	900	72.2	10,550	12.8	2.83	.68	23.1
375	900	76.9	10,700	10.1	2.06	.41	19.6
375A	800	71.0	9,400	15.4	2.50	.94	25.3
375A	900	70.7	10,550	14.3	3.09	.73	25.4
375B	800	72.1	9,100	15.2	2.63	.90	29.8
375B	900	71.8	10,200	13.2	3.04	.57	23.2
378	900	78.1	10,300	7.9	2.14	-	20.3
378A	800	71.2	9,050	15.5	2.76	-	28.2
378A	900	71.0	10,350	12.9	3.24	-	24.9
378B	900	72.1	10,300	13.0	3.13	-	23.9
High-volatile coals							
346	900	71.8	10,450	14.0	2.50	0.46	18.7
346A	900	70.0	10,500	14.5	2.63	.55	22.7
346B	900	70.7	10,500	13.8	2.59	.53	19.2
348	800	69.5	9,050	15.5	2.08	.72	27.4
348	900	69.4	10,200	13.8	2.80	.46	23.3
348	1,000	69.3	11,400	11.9	2.74	.29	18.0
352	900	64.3	10,750	14.7	2.70	.68	25.0
356	800	67.4	8,900	17.1	1.57	1.10	26.4
356	900	67.0	10,300	14.6	2.15	.90	25.7
356 4/	870-1,010	67.1	10,200	11.6	1.60	.59	26.0

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TABLE 9. - Yields of carbonization products, as-carbonized basis (Cont'd.)

Coal No.	Carbonizing temperature, °C.	Coke, percent	Yields per ton of coal[1]: Gas, cubic feet	Tar, gallons	Light oil, gal. In gas	Light oil, gal. Tar to 170° C.	$(NH_4)_2SO_4$, pounds
			High-volatile coals				
356A	800	71.3	8,850	13.8	2.33	1.10	26.6
356A	900	70.7	10,250	12.7	2.08	.61	22.7
361	800	65.4	8,650	15.4	1.66	1.20	32.1
361	900	64.7	9,950	12.0	1.89	.65	28.7
361[4]	870-1,010	65.7	9,250	9.0	1.80	.69	28.1
361A	800	69.7	8,950	10.2	1.73	.62	29.0
361A	900	68.2	9,450	11.4	2.05	.59	26.1
361A[4]	870-1,010	70.8	9,350	8.4	1.73	.45	25.6
362	800	69.1	8,900	17.9	2.82	1.74	22.0
362	900	69.2	10,200	14.8	2.88	1.22	19.5
362A	800	72.8	8,800	15.3	2.56	1.45	20.9
362A	900	72.4	10,100	13.2	2.79	.99	20.0
362B	800	74.4	9,050	13.6	2.16	1.10	21.1
362B	900	74.1	10,350	11.3	2.39	.70	19.0
362C	800	72.7	9,000	14.8	2.49	1.17	22.6
362C	900	72.3	10,200	14.1	2.74	.89	19.1
362D	800	72.8	8,800	12.9	2.57	.71	22.9
362D	900	72.4	10,100	12.6	2.67	.89	17.4
362F	800	72.5	8,800	14.1	2.69	.90	22.2
362F	900	72.0	10,100	12.5	2.76	.84	17.9
363	800	68.4	9,200	18.5	2.60	1.47	24.3
363	900	68.2	10,500	15.9	2.97	1.19	21.7
363[4]	870-1,010	67.4	10,150	13.3	1.97	.96	22.4
363A	900	72.2	10,000	13.2	3.00	.66	22.7
364	800	66.6	8,800	12.5	2.62	.80	31.2
364	900	66.1	9,900	10.4	2.69	.71	23.2
364	1,000	66.0	11,300	7.9	3.01	.38	14.1
366	800	66.4	8,750	14.7	2.64	1.29	25.8
366	900	65.7	10,100	11.8	3.37	.90	22.1
366	1,000	65.6	11,000	10.1	3.35	.40	19.1

1/ Coke, tar, ammonia, and light oil are reported moisture-free; gas is reported as stripped of light oil and saturated with water vapor at 60° F. and under a pressure of 30 inches of mercury.
2/ Moisture in coal, 6.1 percent.
3/ Moisture in coal, 1.4 percent.
4/ Carbonized in 500-pound slot oven.

Low-Volatile Bituminous Coals

The carbonizing properties of low-volatile coals from four beds in West Virginia were determined.

Davy Sewell-bed coal (345) from Twin Branch mine, McDowell County, was blended with 80 percent Pittsburgh-bed coal, and the blend (345A) yielded 72.3 and 71.7

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percent coke at 800° and 900° C., respectively. This low-volatile coal expanded 11.2 percent in the sole-heated oven, and its blend contracted 3.7 percent. These results indicate that the coal is suitable for blending with high-volatile coals for the production of metallurgical coke.

Pocahontas No. 3 (357) from Lake Superior No. 3 mine, McDowell County, coked strongly at 900° C., either singly or when blended with 80 percent Eagle (362) or No. 2 Gas (363). This coal contained 84.0 percent fixed carbon on the dry, mineral matter-free basis, and it ranks rather high, therefore, in the low-volatile classification. It coked less strongly at 800° C. than other samples from the same bed, probably because of its high rank. It expanded 10.9 percent in the sole-heated oven. Three channel samples from the same mine (a357, b357, and c357) expanded 10.2 to 13.1 percent.

Pocahontas No. 4 (358) from Lake Superior No. 4 mine, McDowell County, coked strongly at 800° and 900° C. Its blend (362F) with 80 percent Eagle (362) yielded coke of metallurgical grade, although it should be carbonized commercially at moderate charge densities because it expanded 3.8 percent. The carbonization sample expanded 16.2 percent, whereas three samples (a358, b358, and c358) from different parts of the mine expanded 20.4 and 25.3 percent in the sole-heated oven. A mixture of equal parts of these two Pocahontas coals proved satisfactory for blending with 70 and 80 percent of mixed high-volatile Eagle and No. 2 Gas coals.

Three samples of Pocahontas No. 6 coal - two (347 and 369) from the Black Eagle mine, Wyoming County, and one (370) from the Louisville mine, Mercer County - coked strongly when blended with 80 percent Pittsburgh coal. Samples from the two mines expanded 5.7 and 6.4 in the sole-heated oven, and their blends with Pittsburgh coal contracted 4.0 and 4.8 percent. These results indicate that the blends containing Pocahontas No. 6 from either mine could be carbonized safely in commercial ovens, and that the coke would be suitable for blast-furnace use.

Medium-Volatile Bituminous Coals

Two medium-volatile coals, both from Clearfield County, Pa., were carbonized singly at 900° C. and in blends with 70 and 80 percent Pittsburgh at 800° and 900° C.

Lower Kittanning or B Miller (374), coal from Springfield No. 4 mine yielded strong coke when carbonized singly, and its blends also coked strongly. It expanded 6.7 percent in the sole-heated oven, and its blends with 80 and 70 percent Pittsburgh contracted 4.3 and 0.7 percent, respectively.

Upper Kittanning (375) or "C" coal from Springfield No. 6 mine and its blends with Pittsburgh coal coked similarly to Lower Kittanning (374) and its blends. The upper bed, however, expanded much more - 18.4 percent in the sole-heated oven, although its blends, containing 20 and 30 percent Pittsburgh, contracted more (5.6 and 2.0 percent) than the corresponding blends of the lower bed.

Both of these Kittanning coals could be blended with lower-rank coals for the production of metallurgical coke.

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High-Volatile Bituminous Coals

The high-volatile coals represented 10 beds in five States, as follows: Pennsylvania, 3; West Virginia, 4; Kentucky, 1; Illinois, 1; and Colorado, 1. All ranked as high-volatile A except Illinois No. 5, which ranked high in the high-volatile B classification.

Pittsburgh coal (348) from Republic Banning mine, Fayette County, Pa., was tested under a cooperative agreement primarily to determine the yields obtainable at 800°, 900°, and 1,000° C. from the unblended coal. The 900° C. yields were: Coke, 69.4 percent; and on the basis of per ton of coal, gas, 10,200 cubic feet; tar, 13.8 gallons; light oil, 3.26 gallons; and ammonium sulfate, 23.3 pounds.

Upper and Lower Freeport-bed coals from Kent No. 1 and Kent No. 2 mines, Indiana County, Pa., were blended in approximately equal proportions at the mines; therefore, they were tested as one coal (346). The 900° C. coke resisted breakage well in shatter test, but was rather abradable in the tumbler test. Blending with 30 and 70 percent Pittsburgh coal of similar rank weakened the coke slightly.

Eagle coal (362) from Cannelton No. 3 mine, Fayette County, W. Va., coked strongly; the cokes were significantly stronger than average cokes from high-volatile A coals. Two blends (362D and 362F) containing 80 percent Eagle and 20 percent Pocahontas No. 3 (357), or 20 percent Pocahontas No. 4 (358), respectively, yielded similar cokes, which were but slightly stronger than coke from 100 percent Eagle. Previous investigations have shown that the increase in coke strength gained by blending an exceptionally strongly coking high-volatile coal, like Eagle, with low-volatile coal is small.

No. 2 Gas coal (363) from Cannelton No. 100 mine, Kanawha County, W. Va., yielded coke that was weaker than the average from high-volatile A coals. However, when it was mixed in equal proportions with Eagle (362) for blending with low-volatile coal the resulting blends (362A, 362B, and 362C), containing 35 or 40 percent No. 2 Gas, coked about as strongly as similar binary blends (362D and 362F) composed of Eagle and Pocahontas coals. It was concluded, therefore, that No. 2 Gas may be substituted for half of the Eagle in a blend without appreciable loss in coke strength. A blend (363A) of 80 percent No. 2 Gas and 20 percent Pocahontas No. 3 (357) yielded 900° C. coke having shatter indexes equalling and tumbler indexes slightly lower than the coke from a similar blend containing 80 percent Eagle (362). No. 2 Gas contracted 13.3 percent in the sole-heated oven, and its blend (363A) with 80 percent Pocahontas No. 3 expanded 0.7 percent.

No. 5 Block-bed coal (364) from Northland mine, Wayne County, W. Va., yielded coke that withstood breakage in the shatter test but was abradable. The 1- and 1/4-inch tumbler indexes, 24 and 52, respectively, of the 900° C. coke were well below average for high-volatile A coals. The yield of tar (10.4 gallons per ton at 900° C.) was low, indicating that the sample may have been taken from a part of the bed exposed to oxidation.

Stockton-Lewiston-bed coal (366) from the H. G. Jones mine, Lincoln County, W. Va., coked more strongly than No. 5 Block (364), although the low tumbler indexes (1-inch, 34; and 1/4-inch, 53) indicate high abradability for the 900° C. coke. The yield of tar at 900° C. (11.8 gallons per ton) was 2.1 gallons less than the average for 32 coals of similar rank.

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Imboden-bed coal (356) from Shadyside mine, Letcher County, Ky., was carbonized by the BM-AGA and slot-oven methods. The cokes were rather fingery and fractured readily in the shatter test. Blending with 20 percent Pocahontas No. 3 (75) strengthened the coke significantly. Tumbler- and shatter-test indexes of the blend cokes indicate that Imboden coal could be used in the production of metallurgical coke if properly blended. Although Imboden contracted 1.5 percent in the sole-heated oven, the blends (356A and 356B) containing 20 and 30 percent Pocahontas No. 3 should be carbonized at moderate bulk densities, because they expanded 6.5 and 8.3 percent, respectively.

Illinois No. 5-bed coal (361) from Sahara No. 16 mine, Saline County, Ill., ranked as high-volatile B, although it was high in that rank. It was carbonized in BM-AGA retorts at 800° and 900° C. and in the 500-pound slot oven. The cokes were highly fractured, and large proportions broke during the physical tests. Blending with 20 percent Pocahontas No. 3 (75) strengthened the coke appreciably. For example, in testing the slot-oven coke, the 1-1/2-inch shatter index was raised from 71 to 89, and the 1-inch tumbler index increased from 29 to 52; the hardness factor or 1/4-inch tumbler index decreased from 68 to 61. No. 5 coal contracted 3.1 percent in the sole-heated oven. Because its blends (361A and 361B) with 20 and 30 percent Pocahontas expanded 1.3 and 3.2 percent, respectively, they should be carbonized at moderate bulk densities in byproduct ovens.

The sample of coal (352) from core-drill hole No. 26-23, Gunnison County, Colo., contained 5.5 percent ash and 0.6 percent sulfur, as received, and it ranked as high-volatile A bituminous. Although the coke was rather fingery in comparison to coke from eastern coking coals, its size and other physical properties were similar to those of coke made from Lower Sunnyside coal. Results of these tests indicate that this drilled sample coked about as strongly as Lower Sunnyside.

No. 5 Block- and Pocahontas No. 6-bed Coals

An investigation of No. 5 Block-bed coal from No. 5 mine, Raleigh County, W. Va., and of Pocahontas No. 6-bed coal from Birdseye mine, Fayette County, W. Va.,[56] showed that both coals could be used in the production of metallurgical coke if blended with suitable coals of different rank. No. 5 Block, which ranked as high-volatile A coal, yielded strong coke when blended with 30 percent Pocahontas No. 3. Pocahontas No. 6, a medium-volatile coal, coked strongly when blended with 70 percent Pittsburgh-bed coal. Pocahontas No. 6 should be cleaned to lower its ash content, 13.9 percent, and lower the abradability of coke made from its blend. No. 5 Block contracted 21.5 percent in the sole-heated oven at a charge density of 55.5 pounds per cubic foot. Pocahontas No. 6 expanded only 0.2 percent under the same test conditions, although the 1.50 floats, which contained 8.6 percent ash, expanded 7.8 percent.

56/ Davis, J. D., Reynolds, D. A., Brewer, R. E., Wolfson, D. E., and Ode, W. H., Carbonizing Properties of No. 5 Block-Bed Coal from No. 5 Mine, Montcoal, Raleigh County, W. Va., and of Pocahontas No. 6-Bed Coal from Birdseye Mine, Sewell, Fayette County, W. Va.: Bureau of Mines Tech. Paper 711, 1949, 72 pp.

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Paonia, Colo., Coal

The carbonizing properties of coals from 10 beds penetrated in core-drilling hole 5-33 near Paonia, Gunnison County, Colo.,[57] were determined by the BM-AGA method. Only the lowest bed yielded coherent coke and that was too weak to be suitable for use in blast furnaces.

Beckley-bed Coal, Raleigh County, W. Va.

A study of the composition and carbonizing properties of low-volatile Beckley-bed coal from Stanaford No. 1 mine, Raleigh County, W. Va.,[58] showed that coal to be suitable for blending with high-volatile A coal for the production of metallurgical coke. Beckley coal expanded strongly during carbonization but should be safe to use in moderate proportions in commercial ovens.

Blending Tests of Appalachian Coals

Physical properties of 900° C. cokes made by the Bureau of Mines-American Gas Association method from 12 high-volatile A Appalachian coals and their blends with 20 and 30 percent Pocahontas No. 3 were tabulated and compared graphically.[59] These conclusions were drawn: (1) Increases in the coking power of high-volatile A coals effected by blending with Pocahontas No. 3 generally were greatest for coals that yield the weakest coke when carbonized singly, and (2) the coke-strength indexes of the blends of an appreciable proportion of the high-volatile A coals increased only slightly on raising the proportion of low-volatile coal from 20 to 30 percent.

Plasticity of Coals

Plastic properties of 37 coals and 28 blends were determined during the fiscal year. Of the 65 samples, table 6 describes 26 of the coals and the 28 blends. In addition, Pocahontas No. 3 bed, Lake Superior No. 3 mine, McDowell County, W. Va., raw whole coal (d357) and the 10 coals described in table 10 also were tested. All samples were tested by either the Gieseler and/or Davis plastometer methods; two or more tests on the same sample were usually made by each test method. A total of 233 tests - 117 by the Gieseler and 116 by the Davis method - were made.

57/ See footnote 9.

58/ Davis, J. D., Reynolds, D. A., Brewer, R. E., Wolfson, D. E., Ode, W. H., and Birge, G. W. Carbonizing Properties of Beckley-Bed Coal from Stanaford No. 1 Mine, Mt. Hope, Raleigh County, W. Va.: Bureau of Mines Tech. Paper 712, 1949, 38 pp.

59/ Reynolds, D. A., Effects of Blending Pocahontas No. 3 Coal with 12 High-Volatile A Coals: Am. Gas Assoc., May 1949, 5 pp.

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TABLE 10. - Special samples for Lurgi process of complete gasification

Coal No.	Description and source
350	Upper Freeport bed, slope mine, Indiana County, Pa.
a350	Upper Freeport (350), fine-size coal.
351	Upper Freeport bed, Watson mine, Indiana County, Pa.
355	Pittsburgh bed, Federal No. 1 mine, Marion County, W. Va.
360	Pittsburgh bed, Laura Lee mine, Harrison County, W. Va.
367	Carbon-Emery bed, Hiawatha mine, Carbon and Emery Counties, Utah.
368	Pittsburgh bed, Jamison No. 9 mine, Marion County, W. Va.
368-h	Pittsburgh (368) preheated at about 400° C. to destroy coking.
368-Ox	Pittsburgh (368) oxidized for 1 hour at 250° to 300° C.
396	Low-ash Kentucky coal preheated at about 400° C. to destroy coking.

The plastic properties of the 26 coals and 28 blends described in table 6 and of Pocahontas No. 3 raw whole coal (d357) were determined in connection with the BM-AGA survey of their carbonizing and expanding properties. As related to these properties, the plastic characteristics may be discussed conveniently according to characteristic temperature indications of certain stages in the fusion range, the degree of maximum fluidity (dial divisions per minute) developed in the Gieseler test method, and the maximum resistance (pound-inches) observed in the Davis test method. In general, as the rank of the coal increased from high-volatile A to low-volatile bituminous, the fusion is displaced to a higher temperature, the maximum fluidity becomes less, and the maximum resistance becomes greater. These changes in plastic properties usually correlate well with corresponding increases in strengths of the cokes produced at 800° and 900° C. from coals of the respective ranks. Variations in chemical and petrographic composition among individual coals of a given rank have a greater influence on the maximum fluidity and maximum resistance values than on the temperature range of fusion. For this reason, the plastic properties of the coals and blends are given in table 11 in terms of these values in relation to the strengths of their corresponding cokes.

Table 10 gives the source of 10 coals whose fusion properties were determined by the Davis plastometer method to ascertain the suitability of these coals for complete gasification by the Lurgi process. The first seven samples listed in the table gave Davis maximum resistance values ranging from 7.8 for coal 360 to 40.5 for coal a350. The last three samples were tested with the new drum-rabble assembly in the Davis plastometer retort and gave Davis maximum resistances of 75.5, 46.8, and 29 pound-inches, respectively. The plastic properties of all 10 samples indicate that these coals would have to be preheated or oxidized for some time before they would be suitable for charging into the Lurgi generator.

Past experience has indicated that the plastic properties of certain coals of poor fusion properties were not evaluated properly by Davis plastometer test method. Such coals tend to fuse around the shaft, and little or no fused material remains between the faces of the rabble arms and the retort wall; the indicated resistance, therefore, is too low. To remedy this defect, a closed drum was built around the shaft, and the four rabble arms were fastened on the outside surface of the drum at the same relative positions as in the old Davis plastometer. Because of the reduced volume in the new assembled retort, a smaller coal sample was required. Numerous tests with 12- and 8-gram samples showed that the 8-gram sample of through 20-mesh coal, as compared with the 18-gram sample used in the old assembly, was suitable.

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Characteristic temperatures, appropriately corrected for the different heat gradients in the two retort assemblies, agreed well. As expected, the Davis maximum resistance obtained on a given coal by the new-type assembly is higher than that by the old assembly because of reduced free volume within the retort.

TABLE 11. - Plastic properties of coals and blends

Coal No.	Gieseler 1/	Davis 2/	Remarks
			Low-volatile coals
345	405	23.2	The plastic properties of these coals followed closely the same order as their relative rank.
370	2,160	40.5	
347	61	60	
358	140	28	The plastic properties of the eight coals and the four blends indicate that if the coals are properly blended, strong cokes may be expected.
369	115	3/	
372	4	4/77	
357	1.6	3/	
d357	4.7	3/	d357 is raw whole coal from the same source as 357.
345A	5/	6/	
370A	5/	6/	
347A	5/	6/	
369A	5/	28	
			Medium-volatile coals
375	900	4/14.5	Plastic properties of 374 and 378 indicate that their cokes should be slightly less abradable than the coke from 375. The appreciable maximum fluidities of the six blends indicate that their cokes would be weaker than the cokes from the corresponding medium-volatile coals.
374	1,580	4/38.5	
378	2,000	4/25	
375B	7/	8/	
378B	7/	8/	
374A	7/	8/	
375A	7/	9/	
378A	7/	9/	
374B	7/	9/	
			High-volatile coals
364	49	3/	The coals tested covered the whole range of rank of high-volatile A bituminous coals (dry, mineral-matter-free fixed carbon less than 69 percent, and moist B.t.u. 14,000 or more). The maximum fluidity, therefore, ranges from very low to peak and then to lower values with increasing rank of the coals. The plastic properties of the high-volatile coals, except for the lower ranking high-volatile A coals and the high-volatile B coal, indicate that they should produce good cokes. Except for blend 346A, which contained two high-volatile A coals and developed a very high maximum fluidity, all other blends should produce strong cokes.
366	97	9.5	
352	106	8.2	
363	30,000	4/12	
363A	1,265	4/19	
346	20,000	28.6	
346A	20,000	31.6	
348	7,845	37	
376	7,500	4/50	
362	5,850	7.5	
362A	5,725	19.3	
362B	1,190	8.8	
362C	2,610	4/79	
362E	320	42.5	
362D	1,000	44.5	
362F	705	4/21	

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TABLE 11. - Plastic properties of coals and blends (Cont'd.)

Coal No.	Gieseler 1/	Davis 2/	Remarks
		High-volatile coals	
o28	5,725	4/25	o28 is Pittsburgh coal containing 62.8 percent dry, mineral-matter-free, fixed carbon and is one of the representative samples of the standard blending coal (28).
356	1,850	8.5	
356A	500	19	
356B	180	27	
373	3,080	4/64	
b371	10/		
c371	10/		
d371	10/		
e371	10/		
f371	10/		
b371A	11/		
c371A	11/		
d371A	11/		
e371A	11/		
f371A	11/		

1/ Gieseler maximum fluidity in dial divisions per minute.
2/ Davis maximum resistance in pound-inches.
3/ Did not fuse at normal rate of 3° C. per minute.
4/ New type plastometer described later in this report.
5/ Higher Gieseler maximum fluidity than for corresponding constituent low-volatile coal.
6/ Lower Davis maximum resistance values than for corresponding constituent low-volatile coal.
7/ Gieseler maximum fluidity value appreciably increased over that of corresponding constituent medium-volatile coal.
8/ Higher Davis maximum resistance values than for corresponding constituent medium-volatile coal.
9/ Lower Davis maximum resistance values (new type plastometer) than for corresponding constituent medium-volatile coal.
10/ Gieseler maximum fluidity values ranged from 2865 for coal f371 to 5500 for coal b371.
11/ With the exception of blend c371A, the Gieseler maximum fluidity values of the blends were higher than those of the corresponding channel samples. Different degrees of fluidity may be expected because the composition of the coal varies from point to point in the mine.

Expanding Properties

Results of the expansion tests are given in table 12. Averages are given for tests in the sole-heated oven except for three coals that were not tested in duplicate. All results are expressed as expansion, or contraction at a bulk density of 55.5 pounds per cubic foot.

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TABLE 12. - Expanding properties of coals

Coal No.	Test moisture percent	Average test bulk density, pounds per cubic foot	Sole-heated oven expansion, percent		Vertical oven, maximum pressure on movable wall, pounds per square inch 2/
			At 55.5 pounds per cubic foot 1/	Dry solid coal	
		Low-volatile coals			
345 3/	1.7	52.70	+11.2	69.0	-
345A	1.6	53.70	-3.7	46.3	-
347 3/	1.4	54.68	+6.4	63.7	-
347A	2.1	53.33	-4.8	45.3	-
357	1.1	55.43	+10.9	68.3	-
a357	1.5	53.07	+13.1	72.6	-
b357	1.4	53.13	+13.1	73.7	-
c357	1.8	53.75	+10.2	68.8	-
358	1.0	53.47	+16.2	74.6	-
a358	1.4	52.99	+25.3	89.1	-
b358	1.5	53.15	+20.4	82.2	-
c358	1.3	53.22	+21.3	85.0	-
370	4.4	53.05	+5.7	59.4	-
370A	2.8	53.22	-4.0	46.5	-
372	2.3	53.56	+25.1	95.3	-
374	1.7				
		Medium-volatile coals			
374	1.7	53.63	+6.7	57.9	-
374A	1.9	53.46	-4.3	44.6	-
374B	2.0	53.45	-.7	50.2	-
375	1.1	52.98	+18.4	72.0	-
375A	1.7	53.29	-5.6	41.2	-
375B	1.6	53.28	-2.0	46.6	-
		High-volatile coals			
b326	3.4	55.27	-10.1	29.1	-
b326	3.4	51.87	-	-	3.6
b326	3.4	21.71	-	-	4.1
a331	3.6	53.07	-5.6	46.3	-
a331	3.6	50.56	-	-	1.1
a331	3.6	50.41	-	-	2.0
346 3/	1.7	54.83	+1.8	44.2	-
346A	1.8	53.05	-16.2	27.5	-
356	3.0	52.70	-1.5	49.4	-
356A	2.7	52.29	+6.5	58.7	-
356B	2.4	53.37	+8.3	62.6	-
361	6.4	53.22	-3.1	57.0	-
361A	5.5	53.06	+1.3	63.7	-
361B	5.2	53.11	+3.2	65.0	-
362	1.9	53.41	-2.6	46.0	-

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TABLE 12. - Expanding properties of coals (Cont'd.)

Coal No.	Test moisture percent	Average test bulk density, pounds per cubic foot	Sole-heated oven expansion, percent		Vertical oven, maximum pressure on movable wall, pounds per square inch 2/
			At 55.5 pounds per cubic foot 1/	Dry solid coal	
		High-volatile coals (Cont'd.)			
362A	1.5	53.31	+1.4	51.4	-
362B	1.6	53.53	+4.8	55.0	-
362C	1.7	53.28	+4.1	53.3	-
362D	1.6	53.41	+5.1	55.0	-
362E	1.6	53.67	+5.1	56.5	-
362F	2.5	53.63	+3.8	54.3	-
363	1.7	53.60	-13.3	29.8	-
363A	1.4	53.25	+.7	51.3	-
364	4.9	53.84	-15.9	31.1	-
366	4.4	53.30	-17.8	35.1	-
371	2.4	53.44	-23.1	22.9	-
b371	2.6	52.79	-11.0	36.5	-
c371	2.5	53.11	-4.4	45.5	-
d371	3.4	53.34	-9.9	43.5	-
e371	4.1	53.02	-15.1	34.3	-
f371	5.3	53.03	-11.2	39.0	-
b371A	3.6	52.43	+1.0	56.5	-
c371A	3.2	52.76	+6.5	59.6	-
d371A	3.7	53.05	-.2	54.8	-
d371A	3.3	49.72	-	-	1.5
d371A	3.4	50.88	-	-	1.9
e371A	3.7	53.00	-3.6	51.8	-
f371A	4.9	53.07	-3.3	52.0	-
373	2.4	56.37	-6.7	46.0	-
376	.8	55.31	-11.9	37.6	-
376	.8	55.46	-	-	3.4
376	.8	54.75	-	-	4.3

1/ End-of-test contraction for contracting coals; maximum expansion for expanding coals.
2/ Each vertical-oven test reported individually.
3/ Single tests.

In a study of Thick Freeport-bed coal (371), six channel samples of "low coal" were blended with 17-1/2 percent Lower Kittanning (372) for expansion tests. "Low coal" is taken from those parts of the mine where the upper and lower benches have merged. Because the lower bench coal normally expands slightly, this "low coal" may be unsafe to blend with the strongly expanding Lower Kittanning coal. Sample c371 contracted the least of the "low coals," and its blends (c371A) expanded the most (6.5 percent). The other blends contracted, except b371A, which expanded 1.0 percent.

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Upper and Lower Freeport coal (346 and b326), Upper Freeport (a331), and Upper Freeport (376) are borderline coals that frequently have yielded cokes that have been difficult to push when carbonized commercially. Upper and Lower Freeport (346) was the only one of these coals to expand in the sole-heated oven, and the fact that another sample (b326) did not expand is not surprising, because Freeport coals usually are inconsistent in expanding. Sample b326 exerted pressures of 3.6 and 4.1 percent during carbonization in the large vertical oven. Two samples of Upper Freeport, representing different mines, differed in expanding properties - Sigley mine coal (376) contracted 11.9 percent in the sole-heated oven and produced wall pressures of 3.4 and 4.3 pounds per square inch in the large vertical expansion oven, whereas Watson mine coal (a331) contracted only 5.6 percent in the sole-heated oven and produced wall pressures of 1.1 and 2.0 pounds per square inch in the large vertical expansion oven.

New Experimental Coke Oven

During the past year the 1/2-ton laboratory coke oven installation at the Bureau of Mines Southern Experiment Station, Tuscaloosa, Ala., was completed. From preliminary tests it was concluded that:

1. The quality of the coke produced in the oven, under identical operating conditions, is reproducible within the known limits of accuracy of the conventional ASTM tests.

2. The quality of coke from the test oven is substantially the same as that from a full-scale commercial oven when the same coal mix and operating conditions are used.

3. The wall pressure-time history for a given coal under the same carbonizing conditions is reproducible.

4. The apparent specific gravity of the coke from the test oven is approximately 10 percent lower than commercial coke. This difference is due to the oven geometry.

5. The test oven is substantially free from end effects when the coke 6 inches from the unheated faces is considered.

6. The addition of 1 quart of oil per ton of coal charged increases the bulk density by 5 percent and the apparent specific gravity of the coke by 2 percent. Additions of greater quantities of oil would cause a further increase in these two quantities.

Oxidizing Properties of Bituminous Coals

The effects of oxidation at room temperature and at 99.3° C. on 11 coking coals were determined.60/ The effects on carbonizing properties were decreased strength and fusion of coke and yield of tar, increased apparent specific gravity of coke and yield of ammonium sulfate, and irregular proportions of acids and residual pitch in

60/ Brewer, R. E., Reynolds, D. A., Steiner, W. A., and Van Gilder, R. D. Carbonizing Properties of Coking Coals, Effect of Oxidation in Storage: Ind. Eng. Chem., vol. 40, 1948, p. 1243-1254.

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tar. The most sensitive measures of extent of oxidation of coal were agglutinating value, yield of tar, and coke strength. Correlation of changes in coal and coke properties of the sample oxidized at room temperature with those of samples oxidized at 99.3° C. established simple factors used to express relative rates of oxidation at the two temperatures.

Carbonizing Properties of Chilean Coals

The carbonizing properties of 10 Chilean coals[61] were determined in an investigation to determine (1) whether metallurgical coke could be made from six high-volatile coals, and (2) whether the quality of four lower-rank coals could be improved. Of two Schwager-mine coals, No. 5 bed was better for coking because it contained less sulfur and coked more strongly than No. 3-bed coal. Of four Lota-mine coals, the Alta bed, Pique Nuevo, had the highest coking power and the most satisfactory composition. These coals contracted during carbonization; they should give no trouble from expansion in coke ovens when blended with as much as 30 percent low-volatile expanding coal. They did not oxidize excessively, as might be expected of coals of similar rank. Four noncoking coals representing (1) Lirquen bed, Lirquen mine, Province of Concepcion; (2) Pupunahue mine, Mulpun, Province of Valdevia; (3) Mina Vulcano, Province of Magallanes; and (4) Mina Elena, Isle de Riesco, Province of Magallanes yielded large volumes of gas at 1,000° C., because much of the inherent water was converted to water gas.

Survey of the Coke Industry

Postwar progress in the coke industry[62] includes new and improved carbonization plants, upgrading of coal, new sources of blending coals, and improved coke evaluation.

In an evaluation of trends in our creosote-oil supply,[63] largely derived from coke ovens, the role of basic domestic production imports and other influencing factors was considered, as were the factors that will influence the future supply of wood-preserving raw materials.

German Low-Temperature Coal-Tar Industry

Members of technical fuel missions to Germany, organized under the auspices of the Technical Industrial Intelligence Committee, have written a great many reports describing German developments in mining, preparation, and utilization of coal. These reports are on file at the Bureau of Mines, Washington, D. C., where they are available for public examination. Because of the importance of the subject material, some of the reports or combination of reports were published.

The production and processing of low-temperature coal tars in Germany were not associated with high-temperature tar operations. Investigation disclosed that Germany's increased demands for liquid fuels during the period between 1933 and 1945 had brought about the development of low-temperature coal carbonization industry

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61/ See footnote 17.

62/ Davis, Joseph D., Coal Industry (Coke Industry): Min. Eng., vol. 1, No. 3, sec. 2, 1949, pp. 120-122.

63/ Brown, Ralph L., Creosote Oil Supply: Proc. Am. Wood Preservers' Assoc., vol. 44, 1948, pp. 246-259.

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which produced more coal tar than the long-established Germany high-temperature industry.64/ Information on the processes in use was obtained on visits to major plants in the American, British, French, and Russian zones of occupation.

GASIFICATION OF COAL AND COKE

Use of Oxygen in Gas Manufacture

Progress on studies of the reactions, which are of interest in oxygen gasification and in the art of producing tonnage oxygen was reported for the Subcommittee on Use of Oxygen in Gas Manufacture of the American Gas Association.65/ Included in the report were statements on the progress of the design, construction, and operation of oxygen plants prepared by leading manufacturers of tonnage oxygen plants.

The Lurgi Process of Complete Gasification

This process is a method for gasifying fuel (char or noncoking coal) in oxygen and steam at 20 to 30 atmospheres pressure. The work is being carried on in the Central Experiment Station, Pittsburgh, under cooperative agreement with the Southern Natural Gas Co., with equipment designed by O. Hubmann of the Lurgi Co. The process is continuous and designed to consume 3,000 pounds of fuel per day (24 hours), the grate area of the present generator being 1 square foot.

Three coals have been tested in the continuous generator. They are: Corona coal from Alabama, Pittsburgh coal from northern West Virginia, and Hiawatha coal from Utah, the latter being weakly coking. The rate of gasification has ranged from 4,500 to 5,000 cubic feet per square foot per hour, the make gas containing about 28 percent CO_2. After scrubbing out the CO_2, the yield is about 57,000 cubic feet of 420 B.t.u. gas per ton of fuel gasified. The fuel used was low-temperature char in all cases, it being impossible to gasify a caking coal in the generator. It was found practical to use virtually all the CO and H_2 in the gas to synthesize CH_4, using a nickel catalyst, and produce a gas of 900 to 950 B.t.u.

Gasification of Lignite and Subbituminous Coal

Research work of the Bureau of Mines on the gasification of lignite and subbituminous coal was reviewed.66/ Two pilot plants were built to develop a retort-type system of gasification - a small plant at Golden, Colo., and a larger commercial-scale plant at Grand Forks, N. Dak. These two pilot plants have demonstrated that lignite and subbituminous coal can be gasified or carbonized at relatively high speed and efficiency.

64/ Rhodes, E. O., German Low-Temperature Coal-Tar Industry: Bureau of Mines Inf. Circ. 7490, 1949, 84 pp.

65/ Newman, L. L., Report of Subcommittee on Use of Oxygen in Gas Manufacture: Proc. Am. Gas. Assoc., New York, 1948, pp. 302-307.

66/ Parry, V. F., Developments by the Bureau of Mines on Gasification of Lignite and Subbituminous Coal: Skillings Min. Rev., vol. 37, 1948, pp. 1, 4, and 15.

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Gasification of Lignite for Preparation of High-Hydrogen Water Gas

Investigations of the gasification of lignite in the externally heated, continuous, annular retort were continued at Grand Forks, N. Dak., during the past fiscal year. Table 13 summarizes this work.

TABLE 13

Run	Retort tube	Hours operated	Lignite used, pounds	Gas made, cu. ft. S.G.C.
9	Cast HK alloy	98	28,400	640,000
10	Cast HK alloy	53	1/	1/
11	Rolled plate 310 alloy.	694	344,760	7,303,773
		845	373,160	7,943,773

1/ Tube failed.

This additional operational experience has contributed to the perfection of the process and equipment. The heart of the gasification unit is the retort tube, and attention has been concentrated on three major points.

The first point deals with the material out of which the tube may be made. Four tubes have been used so far, and their pertinent characteristics have been determined. Table 14 summarizes this information.

TABLE 14. - Comparison of tubes

Tube No.	Material	Construction	Service
1	Metcolized steel	Welded rolled plate	Unsatisfactory.
2	Pluramelt 446	Welded rolled plate	Failed through distortion.
3	HK alloy	Centrifugally cast one-weld seam	Surface too rough weld failed.
4	310 alloy	Welded rolled plate	Satisfactory.

From an operating experience of more than 4,600 hours with four tubes, it is concluded that coated or sandwich-type tubes are unsatisfactory because of blistering and physical distortion due to difference in coefficients of expansion; as-cast tubes are unsatisfactory because of the roughness of the surface and the eccentricity of the tube; rolled-plate construction with the minimum length of welds is desirable; and 310 alloy apparently is satisfactory.

The second major point is the life of the retort tube. Tube 4 has been operated 694 hours with no sign of serious corrosion. In addition, a flame guard of the same 310 alloy has been in service for more than 4,000 hours and is still in excellent condition. There is no sign of mechanical or physical distortion. A very conservation estimate of the life of this tube is 10,000 hours.

The third major point is the determination of limiting operating conditions for this process and this generator. During this fiscal year it has been determined that (1) the minimum operating temperature should be 1,000° F. at the gas off-take to prevent the formation of tars; (2) the maximum operating temperature should be 1,900°

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F. in the combustion chamber to prevent excessive corrosion and mechanical and physical deformation; and (3) the maximum capacity of the retort is 375,000 cubic feet of dry gas per day, determined by the area of the gas off-take.

Underground Gasification

The gases produced by this process offer a potential source of energy for electric-power generation and for raw materials for synthetic liquid-fuels manufacture. The method holds promise of utilizing coal veins now difficult or uneconomic to mine or recovering the energy yet remaining underground in regions where only part of the coal has been mined out. This latter possibility would entail burning coal pillars that have been left underground in old, worked-out operations.

Laboratory-scale experiments were carried on at Morgantown, W. Va., and the construction and operation of the second field-scale project was undertaken at Gorgas, Ala.

Laboratory Experimentation

In a horizontal rectangular retort, a simulated segment of a coal bed was placed on a hearth constructed of fire brick and insulating brick.67/ The simulated overburden was composed of fire brick adjacent to the coal segment and covered with a thick layer of diatomaceous earth. Inlet and exhaust openings to the retort were fitted with regenerators and suitable equipment for handling blast air and product gases. During experimentation, the retort was altered repeatedly in size and construction, remaining, however, an essentially horizontal and rectangular unit.

Eleven experiments were carried out with this retort. During the first runs, a gas having a heating value of approximately 50 B.t.u. per cubic foot was produced. Marked improvement in gas quality was noted as experimentation continued. Table 15 gives the results of four of the latter test runs made in this retort. The calorific value of the product gas ranges from 92 B.t.u. to 112 B.t.u. per cubic foot. The gas qualities given are the average for the gas-making parts of the several test runs.

During the test runs, the relationship between length of passage and the concentration of oxygen, carbon dioxide, and carbon monoxide in the blast or product gas was determined. Gas analyses were made after the air-gas had traveled 5, 20, 50, 70, and 95 percent of the length of the channel. These analyses clearly showed, first, the decrease in oxygen and an increase in carbon dioxide; then, a decrease in carbon dioxide and an increase in carbon monoxide as the percentage of travel through the channel increased. The 5 percent point was characterized by high oxygen, the 20 percent point by high carbon dioxide, and the 50-, 70-, and 95-percent points by increasing percentages of carbon monoxide with a maximum percentage of carbon monoxide at the 95-percent point. The laboratory tests indicated that it should be possible to use a simple air blast and produce a gas with a heating value of approximately 100 B.t.u. per cubic foot by the underground gasification process.

Second Field-Scale Experiment at Gorgas, Ala.

This second field-scale experiment in underground gasification at Gorgas, Ala. is being conducted in the Pratt coal bed in an area isolated from the main bed of

67/ See footnote 6.

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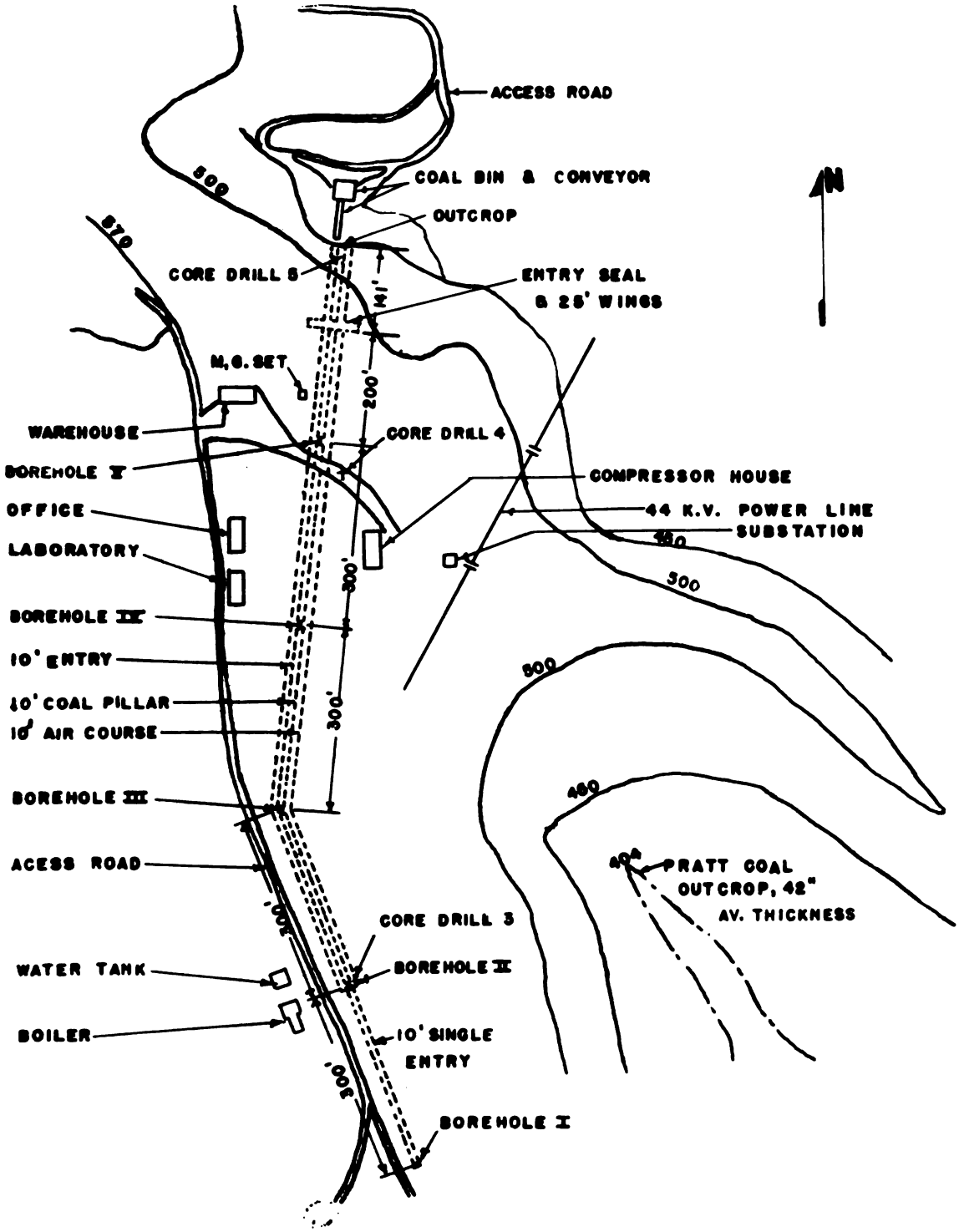

Figure 19. - Plan of underground gasification project.

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coal by nature and consisting of approximately 100.68/ 69/ The coal bed is 42 inches thick and relatively level; it lies under an average over-burden of 150 feet. One of the purposes of this second experiment at Gorgas is to study the characteristics of a unit installation for underground gasification. Such a unit consists of (1) a gasification chamber that is a passageway in the coal bed; (2) inlet and exhaust channels for blast and product gases, respectively; and (3) machinery and piping necessary to operate the system. The installation for the second experiment at Gorgas, Ala., is shown in the schematic diagram (fig. 19).

TABLE 15. - Results of laboratory experiments

Experiment	7	9	10	11
Weight of charge ..... lb..	5,180	8,738	8,185	8,030
Weight of residue ..... do..	1,453	4,920	4,717	3,002
Percent consumed	72	44	43	75
Maximum air flow ..... cu. ft. per hr..	2,350	3,642	4,600	2,940
Analysis of charge, percent:				
Hydrogen	4.6	4.2	4.2	4.5
Carbon	75.9	75.6	78.2	76.6
Nitrogen	1.2	1.3	1.3	1.4
Oxygen	6.1	5.6	4.7	6.2
Sulfur	.9	1.3	.9	.9
Ash	11.3	12.0	10.7	10.4
Heating value ..... B.t.u. per lb.	13,220	13,010	13,390	13,250
Length of run ..... hr..	109.25	83.5	46.3	124.5
Gasification period ..... do..	31.5	52.0	20.3	22.5
Average gas analysis, percent:				
CO_2	8.7	10.2	7.7	7.7
Ill	.2	.6	.5	.3
O_2	.2	.2	.5	.4
CO	19.9	12.1	14.7	20.9
H_2	4.3	9.5	10.7	4.5
CH_4	1.0	2.4	2.0	2.3
N_2	65.7	65.0	63.9	63.9
Heating value ..... B.t.u. per cu. ft..	92	106	112	111
Cold-gas efficiency ..... percent.	55.6	75.0	80.9	64.5

The experimental unit consists of a straight-line passage in the coal bed with surface connections at one end through an outcrop seal and at various points along its length by vertical boreholes. The passage is divided into five sections, four of which are 300 feet long and the fifth 200 feet. Each section may be used separately as a gasification unit, or several sections may be combined to form a major unit. The passage in the coal bed consists of two parallel entries 10 feet wide separated by a solid pillar of coal 10 feet wide. Boreholes have been drilled at 300-foot intervals from the surface to the coal seam, opening at the bottom into crosscuts connecting the two entries. The southernmost 300-foot section consists of a single entry 10 feet wide.

68/ See footnote 6.

69/ Fies, M. H., Elder, J. L., Graham, H. G., Montgomery, R. C., and Jernigan, J. M., The Second Underground Gasification Experiment at Gorgas, Ala.: Descriptive pamphlet of the Gorgas Project, 1949, 19 pp. Issued by Bureau of Mines.

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The objectives of this second field scale experiment are:

1. To determine the quantity of coal that can be gasified from the given initial combustion zone and the shape and extent of the burned-out area formed during this gasification.

2. To test the design and construction of types of product gas outlets, including the seals required.

3. To determine the operational characteristics of the experimental installation under such variation of conditions as the nature of the installation and progress of work indicated to be desirable; for example, the length of passage required, the optimum rate of flow, and the pressure drop.

4. To determine the quality and quantity of the product gas generated under the experimental conditions. A secondary phase of this objective will be the determination of the quantity of tar and related byproducts obtained.

5. To obtain all possible information regarding the action of heat on the overlying strata.

6. To develop, without interfering with the foregoing objectives, such fundamental technical and economic information as is likely to be helpful in selecting plant sites, plant installation, and operating processes.

Construction work on the Gorgas project was started in the latter half of June 1948 and was completed in March 1949. The underground entries were driven into the coal bed, and conveyors were used to load out and transport the coal to the surface. Two 18-inch-diameter and three 28-inch-diameter churn-drill holes were drilled from the surface to the entry. The strata through which each borehole passed were pressure-grouted by means of four 6-inch churn-drill holes spaced 4 feet from the center point of the large hole. Generally, the first two grout holes took appreciable quantities of cement grout, and the last two refused grout, indicating that the underground crevices intersected by the holes were securely stopped.

The five large boreholes were sealed at the surface by lengths of water-jacketed pipe set in concrete (fig. 20). These seals extend approximately 25 feet below the surface and are seated on hard strata. The two 18-inch boreholes are unlined below the surface seal. It is possible that the natural strata will crumble and slag at high temperatures and block these boreholes. To prevent this, the 28-inch diameter holes were fitted with a refractory lining 4 inches thick.

In addition to the large boreholes, a number of holes 6 inches in diameter have been drilled from the surface to the coal bed. Each of these was fitted with thermocouples to measure the temperature rise in the coal bed. The holes are so placed that the progress of the combustion zone may be traced.

An entry seal was constructed across the two entries 141 feet in from the outcrop. This seal consists of a fire-brick wall backed with concrete extending across and 25 feet beyond the outby ribs of the entries. The wall extends 6 feet into top rock and 2 feet into the bottom. Outlet pipes have been sealed into this stopping, so that it may be used to withdraw gas from the underground system.

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Figure 20. - Water-cooled seal installation at borehole V.

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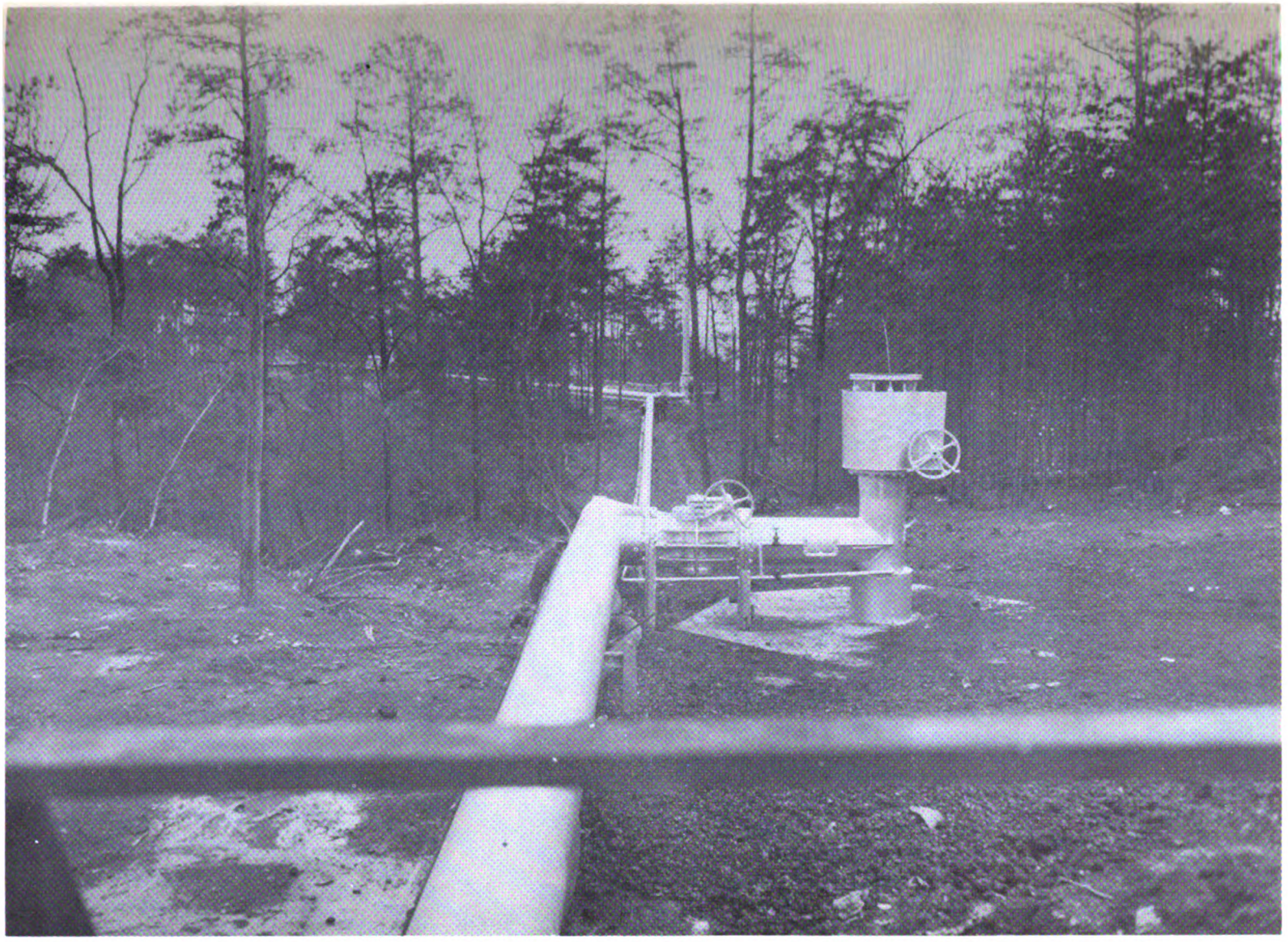

Figure 21. - 20-inch air manifold looking south; borehole IV in foreground.

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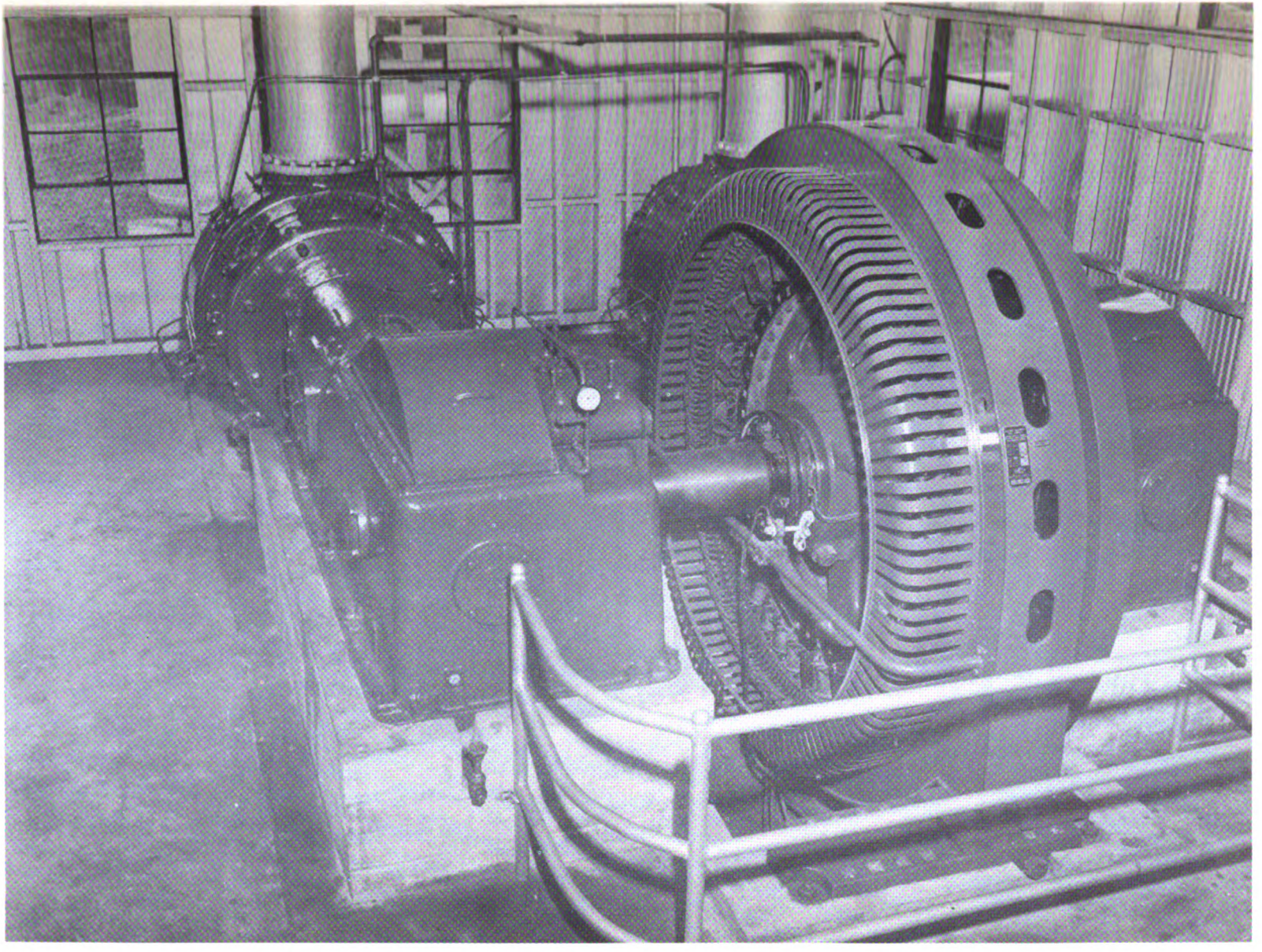

Figure 22. - 800-horsepower reciprocating compressor; capacity, 7,200 c.f.m. free air, 30 p.s.i.g. discharge.

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On the surface, a 20-inch air manifold has been constructed along the line of the entry and extending from borehole I to borehole V (fig. 21). This manifold is connected to each of the boreholes in such a way that the borehole may be used either as an inlet or outlet for the system.

Three electric motor-driven compressors were installed to supply air for gasification of the coal. The major compressor unit is a two-cylinder reciprocating compressor designed to compress 7,200 cubic feet of air per minute with a discharge pressure of 30 pounds per square-inch gage (fig. 22). This machine is driven by an 800-horsepower synchronous motor. Two smaller compressors are for stand-by use.

Operation of the project has been confined to the 300-foot section between boreholes I and II. The ribs of the single entry were undercut to a depth of approximately 15 inches, and a pile of broken coal approximately 15 feet long and 2 feet deep was placed adjacent to the bottom of borehole I. Pine wood was stacked on this pile of broken coal, and the mass of combustible was saturated with fuel oil. On March 18, a thermite hand grenade was tossed down borehole I to ignite the coal seam. At this time air was flowing at a rate of 1,600 cubic feet per minute, entering at borehole I and leaving at borehole II. For several days after the initial firing, temperatures underground continued to rise, the oxygen content of the gases issuing from borehole II decreased, and the carbon dioxide content of the gases increased. After 4 days, the oxygen content in the product gas reached a value of 4.1 percent, and the carbon dioxide content reached 13.6 percent. At this time the carbon monoxide content ranged between 1 and 3 percent. After the fourth day, the oxygen and carbon dioxide contents of the product gases respectively increased and decreased from the values given above until, after 11 days, the carbon dioxide content was 4 percent and the oxygen content 12 percent. Also, during this 11 day period the combustion zone moved from borehole I toward borehole II, and it was then necessary to reverse the flow of blast air in order to drive the combustion zone back toward borehole 1. During these first 11 days the air flow was ranged from 1,500 to 6,000 cubic feet per minute in order to find the optimum flow rate.

After this initial period, the project was operated continuously. Flow was reversed periodically in order to raise temperatures underground to the highest possible level, and an estimated 10 feet of coal has been burned from each of the ribs of the single entry. This quantity of coal is approximately 900 tons. There was no difficulty in maintaining a fire in the coal bed, and there is every evidence that the roof fell uniformly as the coal was burned. The pressure drop over the first 300-foot section of entry increased from approximately 1-1/4 inches of mercury to 12-1/2 inches of mercury with a blast rate of 7,200 cubic feet per minute. Test observations indicate that the pressure drop is very nearly proportional with the length of the entry, indicating a uniform fall of roof. Oxygen is still present in the product gases, but as the resistance to flow has increased the oxygen content has decreased. Good contact between the coal face and the air blast has not yet been achieved, as a by-pass exists through the broken rocks that have fallen into the original entry. A system is being developed whereby this air by-pass through the broken rock will be choked off and the contact between the air blast and the coal face will be improved. At this time the rate of consumption of coal averages 15 tons per day, with a blast rate of 7,200 cubic feet of air per minute. The carbon dioxide content of the product gases ranges between 7 and 8 percent and the oxygen content between 12 and 11 percent. The product gases now always contain carbon monoxide, hydrogen, and methane in amounts ranging from 0.2 to 1.0 percent each. Combustible gases appear to be forming on the coal-coke faces but are being burned by the air by-pass before they can be withdrawn from the system.

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The product gases are being discharged to the atmosphere at temperatures up to 1,100° F. This value can be increased appreciably or decreased at will by varying the length of the cycles. The project is now being operated to store heat underground and thus raise temperature levels there.

The energy derived from coal by underground gasification processes may be utilized either in the form of sensible heat of the product gases or of the heat from combustion of combustible constituents.

A wide choice of operating methods can be applied to this installation. It was originally planned to use a uni-directional air blast; however, as was noted, it was necessary to use a process whereby the blast is reversed periodically. It may be that at a later date this path length will be increased to improve the quality of the product gases or to increase the capacity of the system. Oxygen-enriched air or an oxygen-steam blast have been considered for use in underground gasification and undoubtedly would improve the quality of the product gases obtained; however, fundamental information can be gained from the use of a simple air blast at a much lower cost. Efforts are being made to improve the air blast-coal contact to generally improve the conditions so far obtained on this first section of the project that is being operated.

RESEARCH ON THE PRODUCTION OF SYNTHESIS GAS

Gasification of Pulverized Coal

As a result of substantial improvements made in this simple and easily controlled laboratory-scale synthesis gas process, it is now possible to gasify continuously the most strongly coking coals as well as the cheapest low-grade coal of high ash and sulfur content. The small-scale pilot plant built is particularly well-suited for testing various types of fuels for their synthesis gas-making properties. This is of the utmost importance in designing large-scale equipment, as appreciable differences exist among coals of various ranks and types in completion of gasification, yield of synthesis gas oxygen and steam requirements, etc.

A laboratory-scale pilot unit was designed and a method developed for the gasification of 10 to 50 pounds of pulverized bituminous coal per hour by entrainment in oxygen and steam at generator temperatures ranging from 1,800° to 2,400° F.

A flow sheet (fig. 23)70/ shows the coal-feeding system, the generator chamber, and the following dry residual-dust and fly-ash recovery system. The synthesis gas was then metered, tested, and either stored or vented.

During the first 40 runs, coal up to 30 pounds per hour, pulverized so that 80 to 90 percent passed a 200 mesh-per-inch screen, was fed continuously into the generator from a conical feed hopper in which the coal was kept in a turbulent state by fluidizing nitrogen or natural gas. The charge was moved from the hopper through a vibrated tubular trough and was dropped into the water-cooled generator head at a rate uniform over any given hourly period.

Before each run, the 7-foot long, 6-inch wide, circular generator chamber, whose upper 5-foot length was lined with silicon carbide, was preheated by two natural-gas

70/ See footnote 6.

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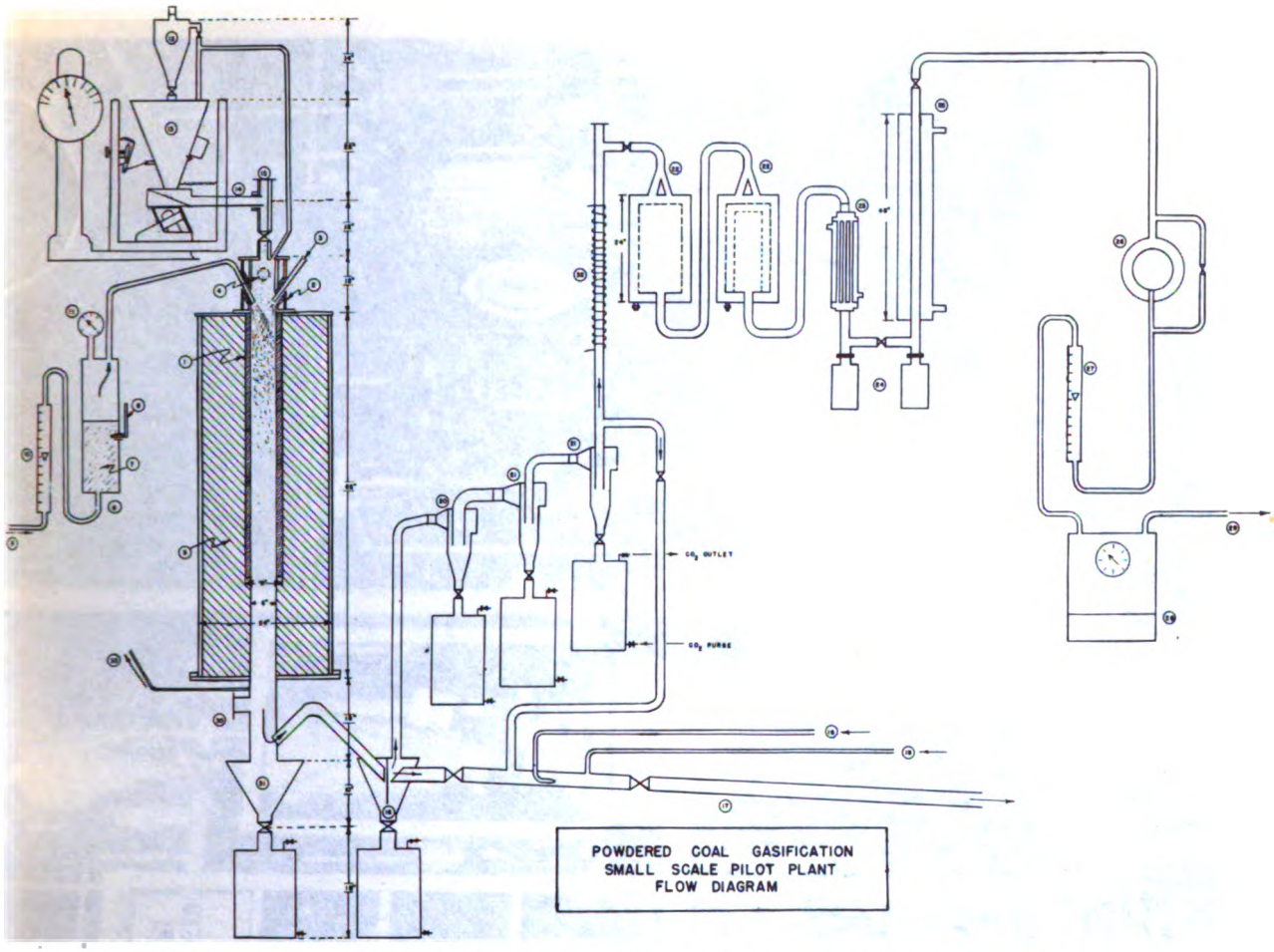

Figure 23. - Flow diagram of small pilot plant for powdered-coal gasification.

1. Silicon carbide generator tube.
2. Water-cooled jackets.
3. Oxygen jets.
4. Burner ports.
5. High-temperature insulating brick.
6. Steam:oxygen ratio controller.
7. Water.
8. Thermometer.
9. Oxygen.
10. Flow indicator.
11. Pressure gage.
12. Charge hopper.
13. Syntron hopper and vibratory feeder.
14. Feed tube.
15. Rupture disk.
16. Knock-out chamber.
17. 2-inch ejector pipe.
18. Steam to jet.
19. Water.
20. 3-inch Aerotec tube.
21. 2-inch Aerotec tube.
22. Fiber-glass filters.
23. Tubular condenser.
24. Condenser water.
25. Secondary condenser.
26. Exhauster.
27. Rotameter.
28. Dry meter.
29. To gas holder.
30. Rupture disk.
31. Slag and residue collector.
32. Heating coil.
33. Gas sample to instruments.

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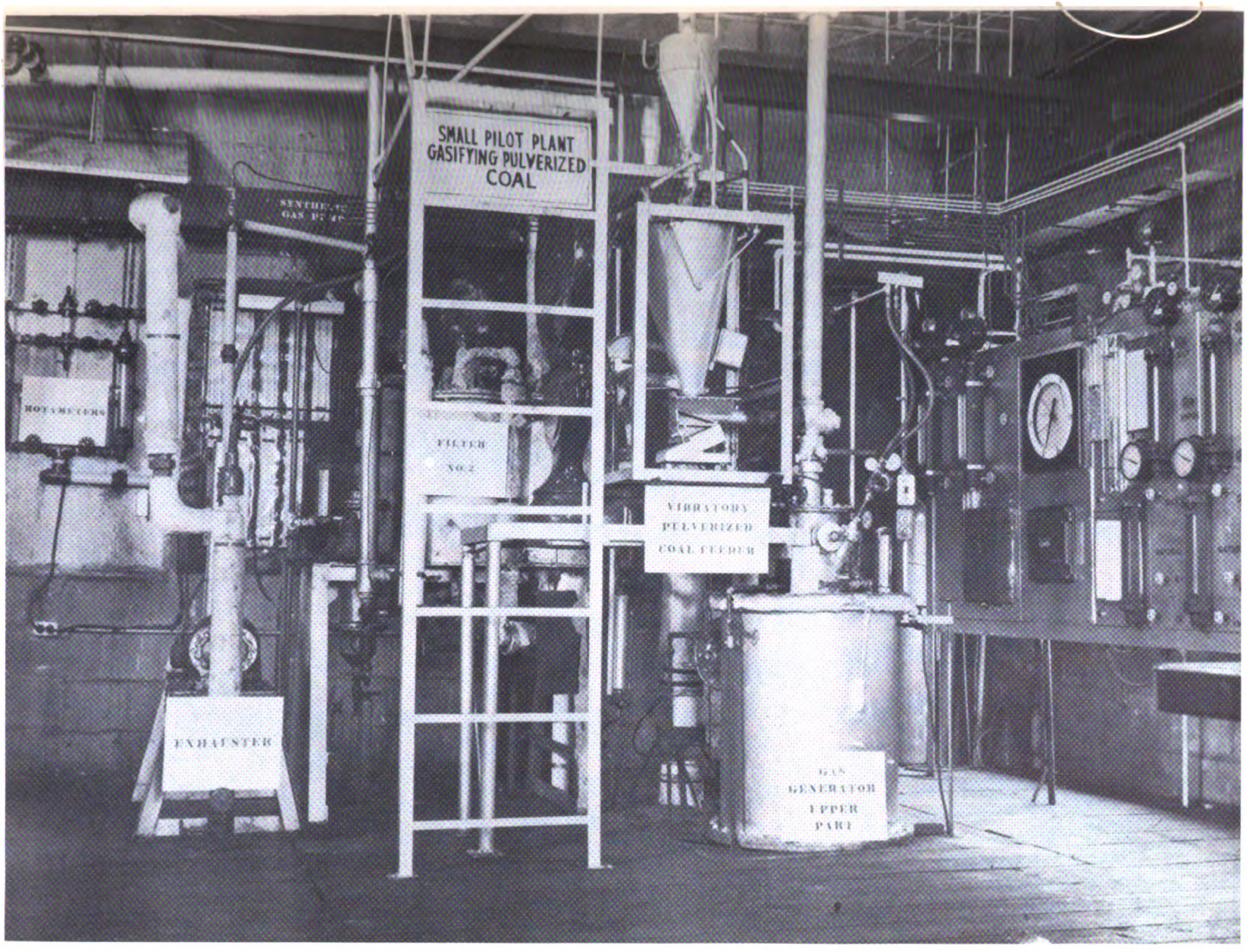

Figure 24. - Upper section of small pilot plant for gasification of pulverized coal at synthesis-gas production laboratories, Morgantown, W. Va.

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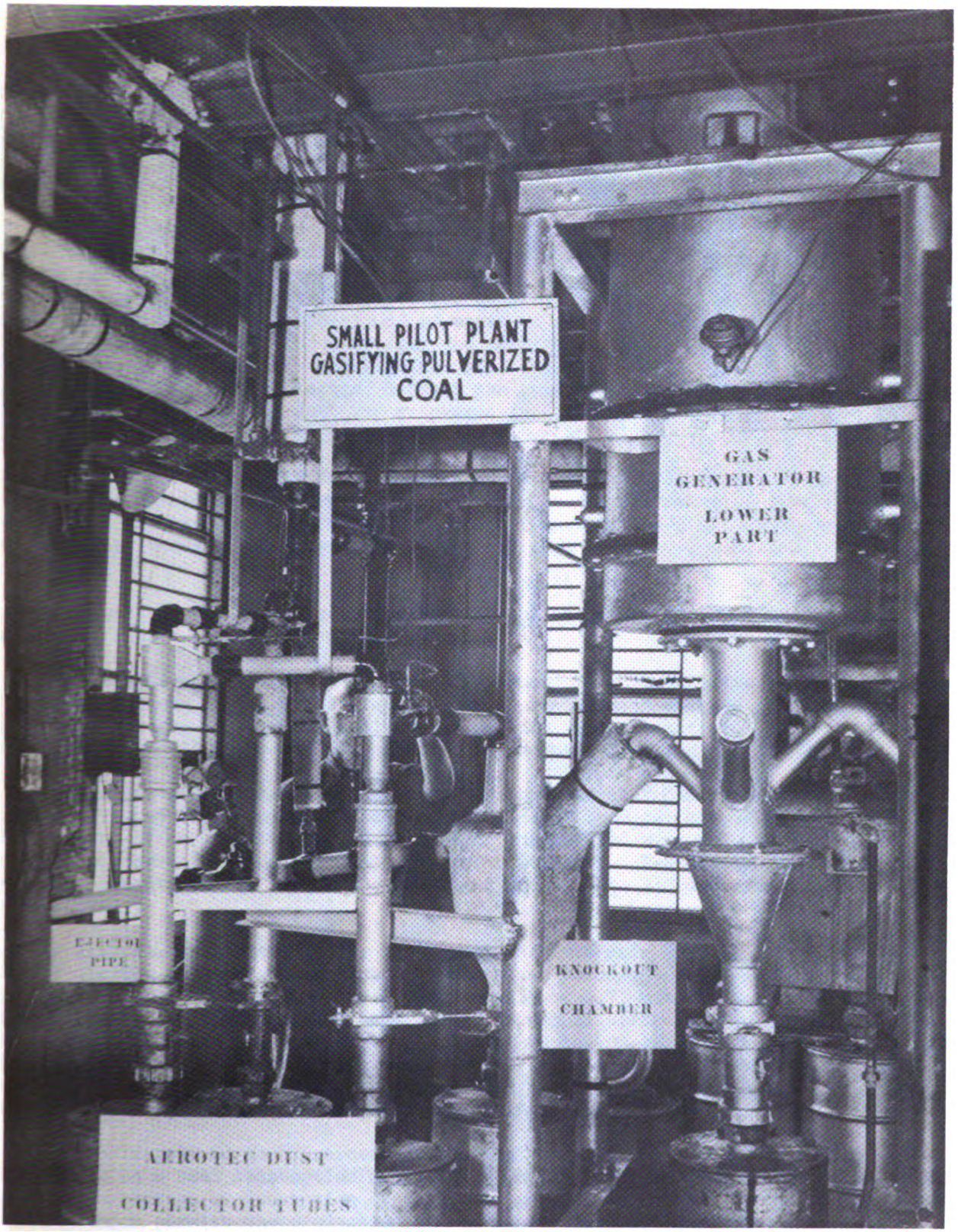

Figure 25. - Lower section of small pilot plant for gasification of pulverized coal.

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flames until the upper half of the lining reached a temperature of about 2,300° F. The run was then begun by shutting the burners off and starting charging of the coal and introduction of oxygen. Steam could be added by passing the oxygen used for gasification through saturating water at constant temperatures, which permitted a close control of the ratio of steam to oxygen.

In the gasification chamber, the fine-coal particles quickly became entrained in turbulent currents resulting from impingement of the two oxygen-steam jets. The carbon in the coal reacted with oxygen to form carbon monoxide and with steam to form carbon monoxide and hydrogen, in addition to minor amounts of other gaseous constituents. Owing to the intentional use of excess carbon and deficiency of oxygen, a certain amount of carbonaceous residue was carried along, together with fly-ash, by the downward-flowing stream of synthesis gas. Coarser particles of slag and residue dropped into the "dust collector" beneath the generator, and the remaining extremely fluffy and light residual dust was recovered in the subsequent units shown in the flow diagram.

The products of gasification could also be discharged through a 2-inch ejector pipe to the outside atmosphere until conditions at the beginning of each run became stable. A constant pressure (up to 3 inches of mercury gage) was maintained in the generator by means of an exhauster, at the end of the train, controlled by a pressure regulator.

Pictures of the upper and lower sections of the small-scale pilot plant are shown in figures 24 and 25.

For the development of the process, a noncoking, high-volatile, bituminous coal from Rock Springs, Wyo., No. 9 seam was used. The coal as charged contained 4.5 percent moisture, 4.9 percent ash, and 37 percent volatile matter, and its ultimate analysis showed 11.9 percent oxygen and 0.8 percent sulfur.

The synthesis gas made in the first 40 runs generally contained 25 to 30 percent of hydrogen, 45 to 50 percent of carbon monoxide, 1 to 2 percent of methane, 0.5 to 1.0 percent of oxygen, 0.2 to 0.9 percent of unsaturated hydrocarbons, 10 to 18 percent of carbon dioxide, and 1 to 2 percent nitrogen. The heating value and specific gravity of the synthesis gas ranged from 240 to 310 and from 0.72 to 0.81, respectively.

A gradual improvement in the quality of the synthesis gas made, i.e., decreasing carbon dioxide content, was observed as the steam-to-coal and oxygen-to-coal weight-ratios were lowered from 0.7 to 0.2 and from 1.4 to 0.7, respectively. In spite of this, owing to unavoidably high heat losses caused by operating the generator below its maximum capacity, the oxygen consumption, 300 to 400 cubic feet per 1,000 cubic feet of "make gas", remained high, as expected. In large-scale production, provided that the generator is run close to its maximum capacity, the consumption of oxygen should be considerably less, probably only half as much.

Approximately 61 to 67 percent of the coal charged was actually gasified, corresponding to a coal consumption of 27 to 33 pounds per 1,000 cubic feet of gas made. The low percentage of conversion of coal into gas was due partly to the use of excess carbon, which permits the production of a synthesis gas low in carbon dioxide and allows a considerable saving in the cost of oxygen. The carbonaceous residue, containing 25 to 30 percent ash, could be recycled and further gasified. However, owing

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to the extremely fine particle size (50 percent less than 7.5 microns) and highly fluffy consistency (bulk density: 2.0 to 2.5 pounds per cubic foot) of the residue, its industrial utilization is not excluded. As shown in figure 26, the particles of the residue approach the characteristic shape, structure, and size of certain commercially used carbon blacks.

The over-all thermal efficiency of the process, i.e., ratio of B.t.u. in coal, was 74 to 82 percent, notwithstanding the high heat losses due to the reasons mentioned. The temperature of the generator was 2,200° F. to 2,450° F. on the top and 1,250° to 1,750° F. at the outlet on the bottom, depending on the combination of several experimental variables. The proportion of steam decomposed was generally 50 percent. The larger pilot plant for pulverized-coal gasification was described 71/ and is shown in figure 27. This plant was completed to the point where gasifying runs were started in the Fall of 1948. To date, approximately 13,000 pounds of coal has been gasified with excellent results at rates ranging from 200 to 350 pounds per hour. Gasification efficiencies of over 85 percent were obtained in these runs, and the thermal efficiency of steam superheating in the range 3,000° to 3,600° F. has been satisfactory.

Sufficient data on refractories have been obtained so that construction of steam superheaters or pebble stoves for continued operation at temperatures up to 4,000° F. can be started soon.

The pneumatic feeder (fig. 28) for pulverized coal has given excellent results in supplying a constant feed of coal to the generator.

A pneumatic feeder 72/ has been developed for feeding pulverized solids to a zone of utilization. This feeder gives promise of solving one of the most difficult problems in conducting heterogeneous reactions, as it provides a method of feeding solids at a uniform rate to a reaction zone. The feeder operates by forming a fluidized bed of solid and gas, from which the solid flows with its carrier gas in dense-phase flow. Most of the fluidizing gas is vented from the top of the apparatus and can be recirculated. A shield around the entrance to the delivery tube evens out the bulk density of the bed, so that the ratio of solid to gas entering the tube is essentially constant. Figure 29 shows a feeder designed to deliver 10 tons per hour of coal to a gasifier operating at 150 p.s.i.g. The carrier gas used in this feeder is synthesis gas.

Studies have been made of the operating characteristics of the feeder, using pulverized coal as the solid and air as the fluidizing gas. It has been found that the coal-air mixture flows through the delivery tube in streamline motion. The viscosity of the mixture is about 30 times that of air alone. The volume-percent of coal in the delivery tube is about 20 percent, and it is controlled by the velocity of the air flowing up through the fluidized bed. Figure 30 shows the calibration curve of the feeder at present installed at the Morgantown pilot plant. This feeder has been found to be quite reliable and accurate.

71/ See footnote 6.

72/ Albright, C. W., Holden, J. H., Simons, H. P., and Schmidt, L. D., Pneumatic Feeder for Finely Divided Solids: Chem. Eng., vol. 56, 1949, pp. 108-111.

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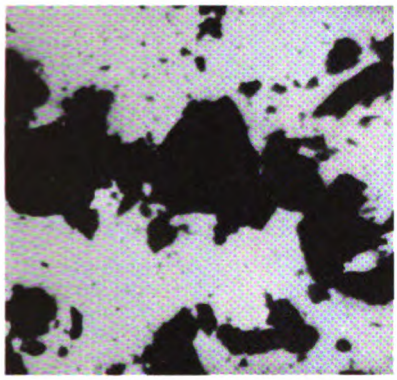
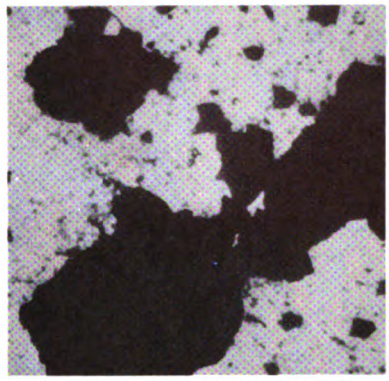
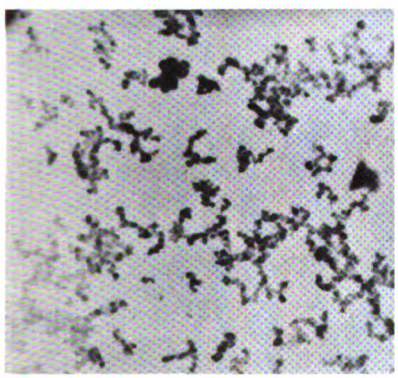


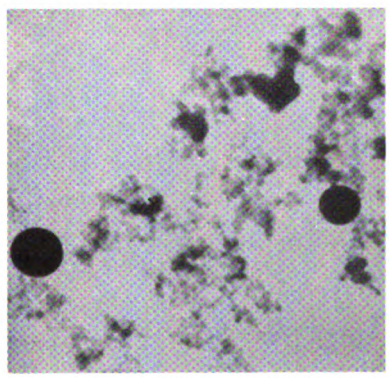
1000 X
Original coal
91% through 200 mesh



1000 X
Carbonaceous residue
from "Dust Collector"



10,500 X
Carbon black
Explosion of acetylene
and natural gas



11,100 X
Carbonaceous residue
from "Dust Collector"

Figure 26. - Electron micrographs of carbonaceous residue obtained in pulverized coal gasification.

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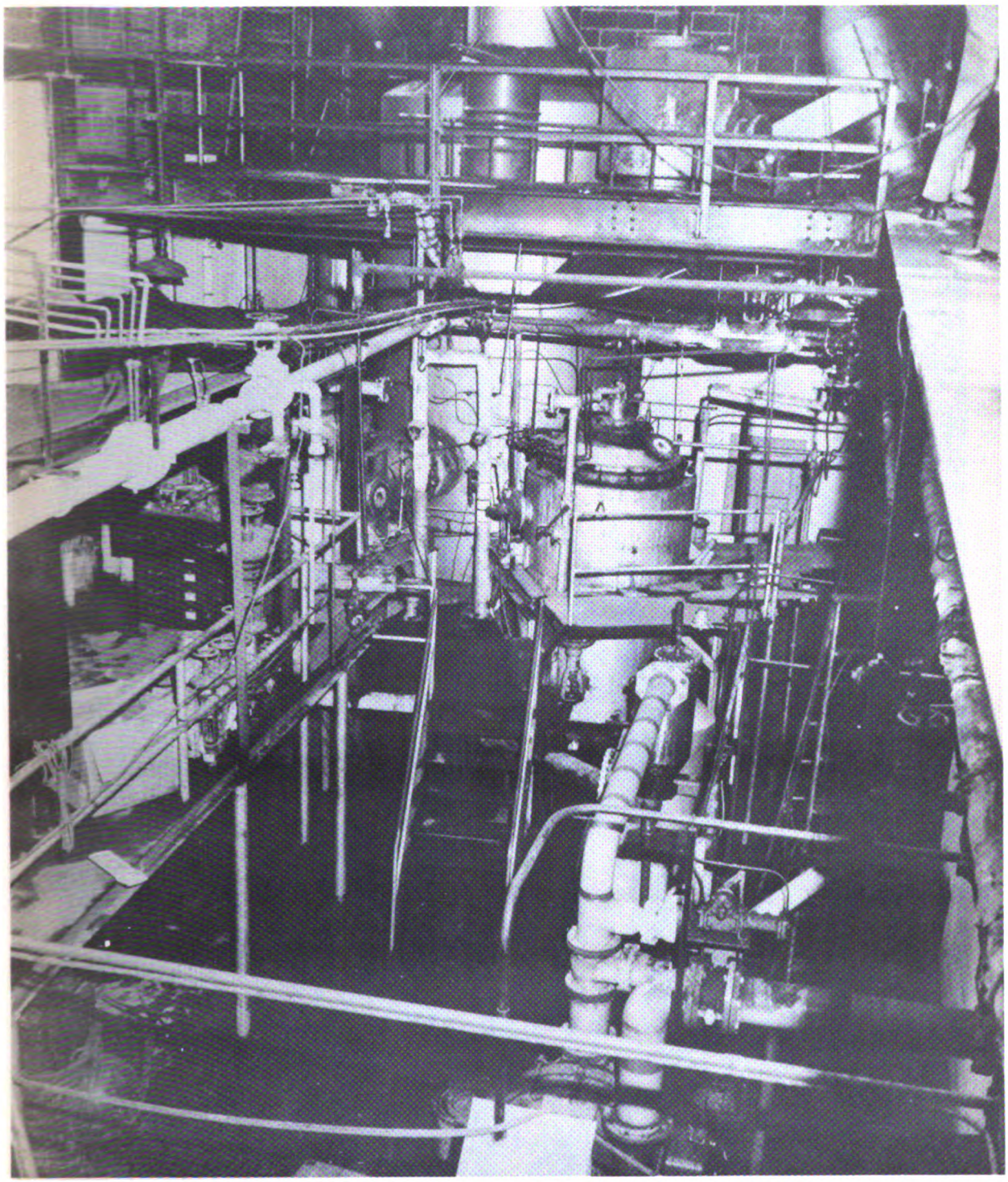

Figure 27. - Inside of pilot plant showing construction work during installation of automatic control equipment, Morgantown, W. Va.

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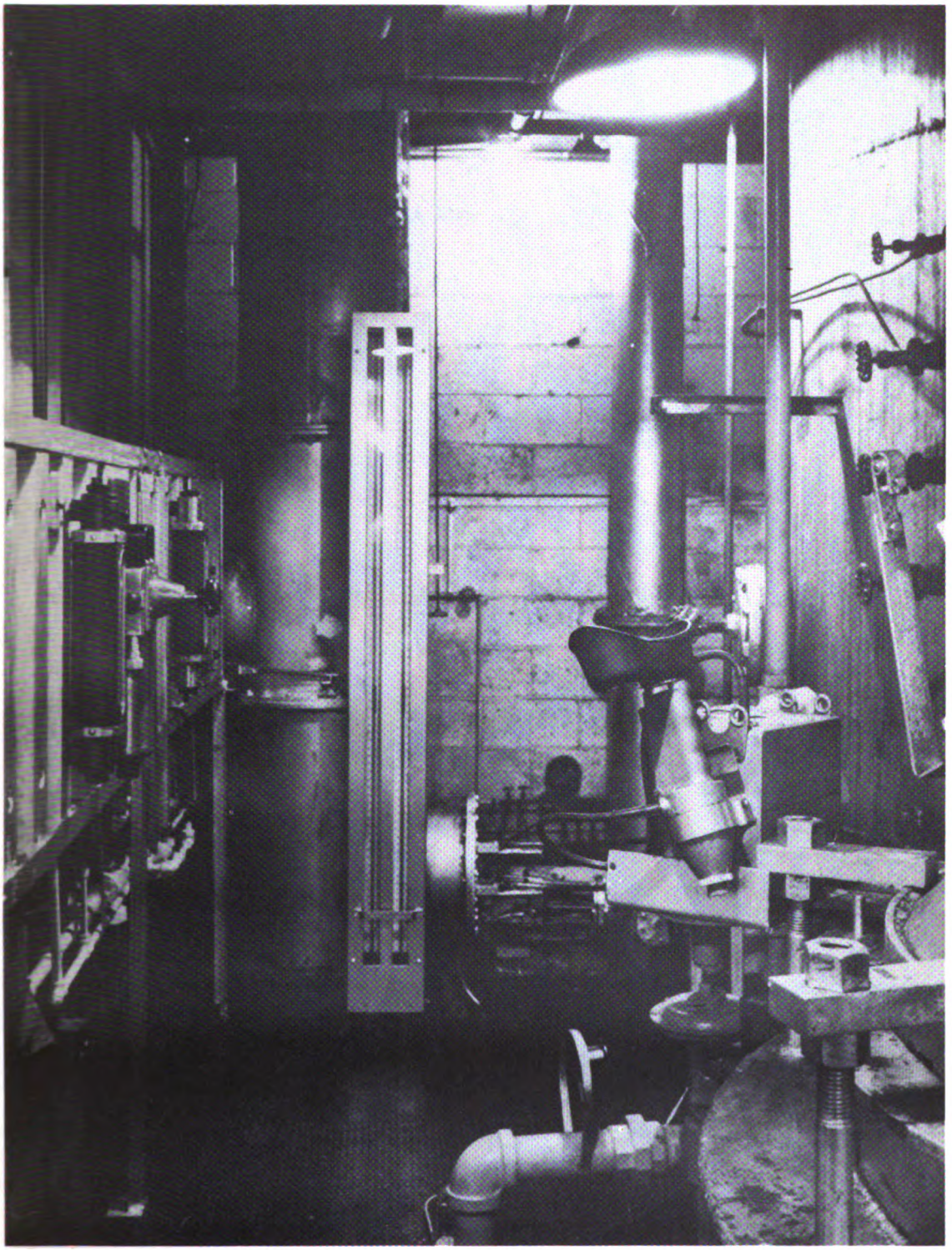

Figure 28. - Pneumatic feeder for charging pulverized coal to gas generator.

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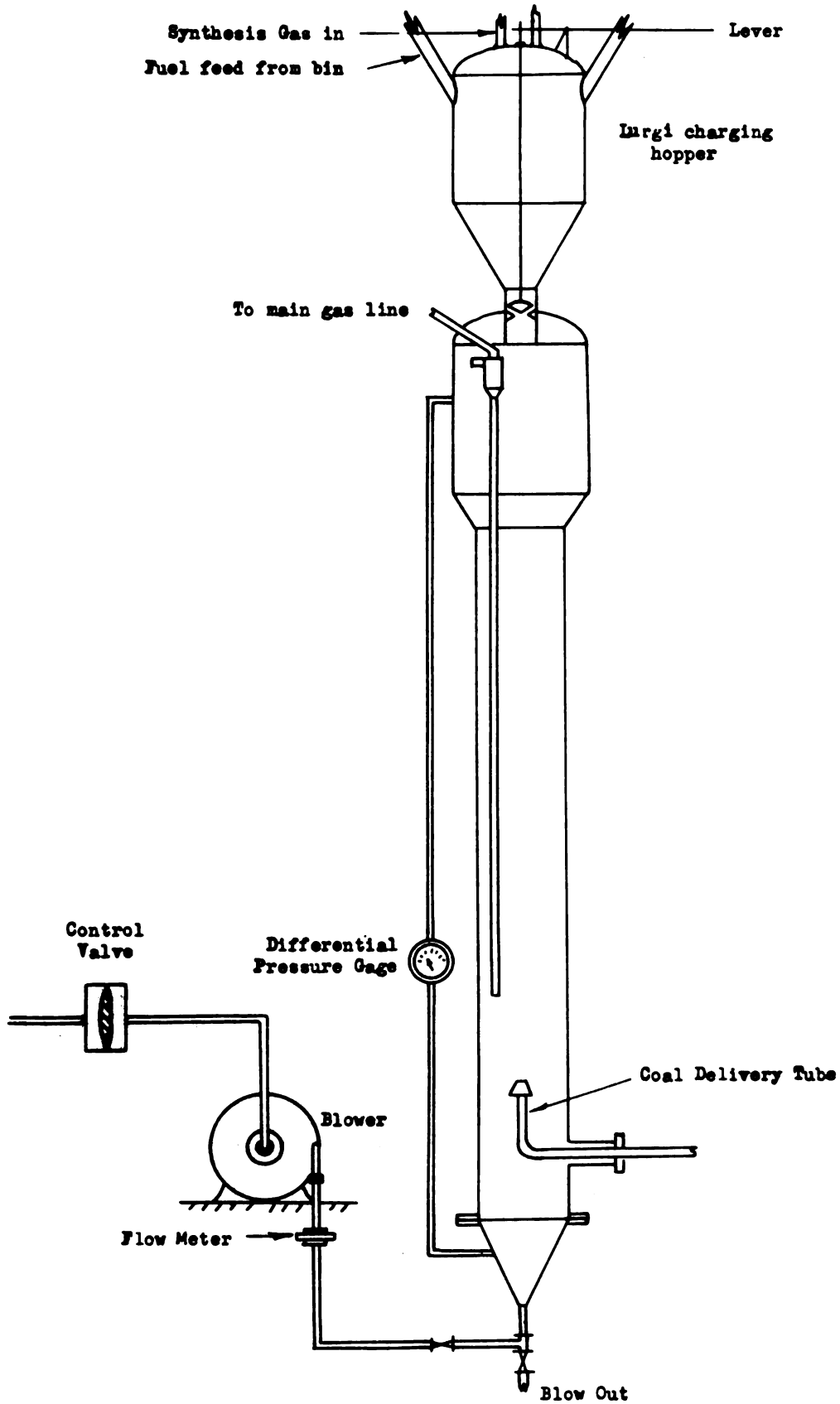

Figure 29. - High-pressure pneumatic feeder.

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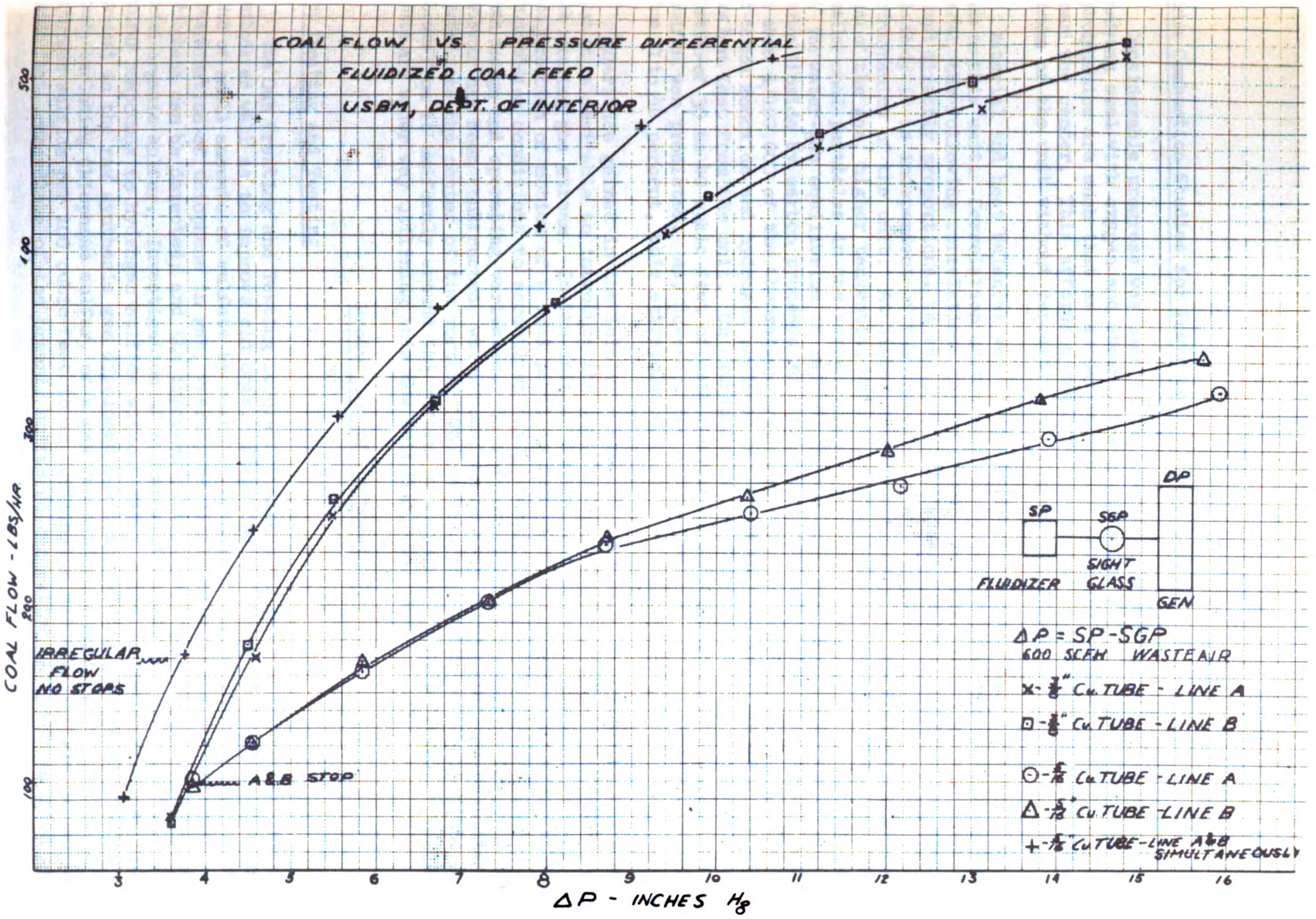
COAL FLOW VS. PRESSURE DIFFERENTIAL
FLUIDIZED COAL FEED
USBM, DEPT. OF INTERIOR
COAL FLOW - LBS/HR
ΔP - INCHES Hg
IRREGULAR FLOW NO STOPS
A&B STOP
SP
SGP
DP
FLUIDIZER
SIGHT GLASS
GEN
ΔP = SP-SGP
600 SCFH WASTE AIR
X - 3/8" Cu TUBE - LINE A
□ - 3/8" Cu TUBE - LINE B
⊙ - 5/16 Cu TUBE - LINE A
△ - 5/16 Cu TUBE - LINE B
+ - 5/16 Cu TUBE - LINE A&B SIMULTANEOUSLY

Figure 30. - Calibration of pneumatic feeder.

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Vortex Combustion Studies for Powdered-Coal Gasification

Considerable progress was made in the operation of an experimental unit for gasifying coal with oxygen and steam. The reactor is of the vortex type, and the hydrodynamics are such that as a coal particle reacts and becomes smaller, it moves toward the center. This affords a means for operating at slagging temperatures in a portion of the unit without deposition of slag on the walls.

Material and heat balances were calculated by using the data obtained from tests made in this unit. Correlation of the results indicates that, for the conditions tested, the following correlations may be drawn: 1. The percent carbon gasified is almost directly proportional to the pounds of oxygen supplies per pound of coal in the feed; it is independent of the throughput, which appears to be a result of the hydrodynamics of the unit. At higher throughputs, despite the shorter residence time of the gas, higher tangential velocities occur in the vortex, thus keeping smaller coal particles in the vortex longer than in the case of lower throughputs. 2. The water-gas equilibrium is reached in the exit gases. 3. The reduction of carbon dioxide by carbon proceeds so slowly that the calculated equilibrium constant is about 1/100,000 of the theoretical value at exit-gas temperatures. 4. The volume of carbon monoxide and hydrogen produced per pound of coal gasified is a function of the heat lost from the unit. On extrapolation of the data to zero heat loss, a production of 30 to 40 cubic feet of carbon monoxide and hydrogen per pound of coal gasified was predicted. As, however, the actual heat losses for this unit ranged from 20 to 40 percent, this extrapolated value must be used cautiously.

Experience gained in the initial stages of operation of the vortex has shown that it is desirable to introduce the coal horizontally, so that it is traveling with the rotating gas stream. In the initial design, the rotating gas streams must change the direction of motion of a coal particle from vertically downward to horizontal. In providing conditions to accomplish this, undesirable limitations are placed upon both design and operating variables. To overcome this difficulty, a new coal distributor has been constructed, which consists of a spinning water-cooled plate inserted into the center of the vortex. The metered coal falls onto this plate and is thrown from it uniformly around the vortex in an umbrellalike pattern. Preliminary tests are under way utilizing this new method of coal feed.

Production of Synthesis Gas

Gas Treating and Testing

The Gas Treating and Testing Laboratory continued to develop new analytical methods as well as to improve old ones. These include the turbidimetric determination of small amounts of sulfate by means of the spectrophotometer; the colorimetric determination of thiophene in gas, whereby as little as 0.0001 grain of sulfur as thiophene can be detected; and the improved platinum spiral method for determining total organic sulfur. Investigation of a number of catalysts for converting organic sulfur compounds to hydrogen sulfide without first removing the hydrogen sulfide present in the raw synthesis gas disclosed one that reduced the organic sulfur content, excluding thiophene sulfur, of a gas containing as much as 700 grains of hydrogen sulfide per 100 cubic feet to an equilibrium value of two grains per 100 cubic feet, regardless of the organic sulfur concentration in the unpurified gas.

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Another type of catalyst being investigated is one that removes hydrogen sulfide and organic sulfur simultaneously by means of catalyst absorption. Whenever the catalyst becomes fouled with sulfur, it is revivified by means of air and steam. Such a catalytic process would eliminate the usual hydrogen sulfide removal system. One catalyst studied removed 99.5 to 100 percent of the hydrogen sulfide and 99.8 to 100 percent of the organic sulfur compounds in the gas.

Oxygen Production by Multistage Fractional Permeation

An important factor in the cost of coal gasification is the cost of oxygen used in the process. At present, oxygen is produced commercially almost exclusively by the Linde process. The fact that many organic membranes are appreciably more permeable to oxygen than they are to nitrogen is the basis for experimental work on a possible process for oxygen production.

Major improvements in the economy of a permeation process could be made by (1) increasing the magnitude of the film permeabilities or (2) increasing the selectivity of the films toward oxygen. Improvements in (1) would permit a decrease in the area of the film required to achieve a given throughput; this might be achieved by finding more permeable films or by making thinner films. Improvements in (2) would not only decrease the size of the plant required but would also decrease the power requirements necessary for a given oxygen production. Such improvements might be achieved by finding films more selective than rubber latex or by incorporating into films specific oxygen carriers (possibly cobalt chelate complexes).

An apparatus was assembled to investigate the selective permeability of organic films, and eight different commercial materials were studied (table 16). Natural rubber is outstanding for its high permeability and its selectivity; however, it is subject to oxidation. Ethyl cellulose and polyethylene are promising. Additional work with ethyl cellulose films cast in the Bruceton Laboratories and with pure gases showed (table 17) that these films had an O_2/N_2 permeability ratio of about 3. Argon is enriched in almost the same proportion as is oxygen, whereas carbon dioxide becomes enriched to a much greater extent. For any commercial application it will probably prove desirable to remove carbon dioxide prior to or just after the first permeation stage. The removal of argon will be impractical, and it will be enriched along with the oxygen.

LIQUEFACTION OF COAL BY DIRECT HYDROGENATION RESEARCH AND DEVELOPMENT

Operation of two coal-hydrogenation pilot plants, one for the first or liquid-phase stage and one for the second or vapor-phase stage, was begun. These plants are concerned with tests on raw materials, catalysts, and procedures to be used in the larger demonstration plant at Louisiana, Mo. New approaches to the problem of direct hydrogenation of coal are being made and are based on the rapid conversion of coal to distillable oil, gas, and coke at moderate pressures, short reaction periods, and relatively high temperatures. The conversion of as much as 50 percent of the coal to coke is not disadvantageous, because about as much coal is needed for fuel as is liquefied in coal-hydrogenation processes. The results of laboratory-scale tests have been encouraging, but the engineering problems are very difficult, and much pilot-plant development work must be done. The chemistry of the primary liquefaction of coal is being studied in extensive laboratory experiments in small high-pressure vessels; some very critical information has thus been obtained.

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TABLE 16. - Film permeabilities to oxygen and nitrogen at 30° C.

Film	Film thickness, mils	Permeability coefficient 1/ Q, cm.^{2} min.$^{-1}$ atm.$^{-1}$ x 10^6		Q_{O_2}
		Q_{O_2}	Q_{N_2}	Q_{N_2}
Ethyl cellulose 2/	0.7	3.25	1.36	2.4
Polyvinyl chloride-acetate	2.0	1.75	1.17	1.5
Vinyl resin - Buna-N rubber	2.0	.70	.58	1.2
Natural rubber latex 3/	7.3	18.1	8.6	2.1
Polyethylene (Plax)	1.5	2.20	.88	2.5
Polyethylene (Bakelite)	.9	2.10	.79	2.7
Polystyrene	1.0	1.31	.44	3.0
Resproid 1404	4.3	3.01	1.08	2.8

1/ Permeability coefficient = (film permeability) (film thickness).
2/ Average of three determinations.
3/ Average of two determinations.

TABLE 17. - Permeabilities of gases through film EC-12

Gas	Permeability (P), 1/ mols x 10^6 cm.$^{-2}$ min.$^{-1}$ atm.$^{-1}$	P_{gas}/P_{N_2}	
		Pure gas studies	Mass spectrometer
N_2 ..	0.075	(1.0)	(1.0)
O_2 ..	.231	3.1	3.2
CO_2 .	1.07	14.3	2/32
A ...	.170	2.3	3.1

1/ Rate of permeation of gas per unit time per unit film area per unit pressure differential.
2/ A blank determination on normal air gave a high CO_2 value.

A study of the composition of synthetic liquid fuels has shown that industrially valuable byproducts of the direct hydrogenation of coal are phenolic compounds used in plastics manufacture. Quantitative analysis of mixtures of these phenols and procedures for separating the components by means of infrared spectroscopic analysis and a counter-current distribution between two immiscible solvents have been developed. The products from the hydrogenation of carbon monoxide consist chiefly of straight-chain, normal hydrocarbons with a minor amount of branded hydrocarbons, the exact isomeric composition of which was determined by infrared and mass spectrometry. This information is of critical scientific importance in ascertaining the mechanism of the synthesis; it is also of industrial significance, because the octane number of the gasoline fraction is markedly dependent on the isomeric composition.

The program for research on coal hydrogenation is indicated in the following outline:

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(A) Function of solvents and catalysts in early stages of coal hydrogenation.

(1) Action of solvents.

(B) Bench-scale process development.

(1) Coal hydrogenation in the absence of solvents.

(2) Substitution of water gas for hydrogen.

(C) Pilot-plant operations.

(1) Liquid-phase hydrogenation.

(2) Vapor-phase hydrogenation.

(D) Separation and identification of products of coal hydrogenation.

Function of Solvents and Catalysts in Early Stages of Coal Hydrogenation

Action of Solvents

The solution of coal by high-boiling compounds that contain both a hydroaromatic ring and an aromatic hydroxyl group (for example, orthocyclohexylphenol) has been described[73] as chemical in nature.

Another type of solvent for coal, as exemplified by phenanthrene, 9-methylphenanthrene, phenanthridine, carbazole, 5,6-benzoquinoline, 5,6-benzoquinaldine, α-naphthol, and β-naphthol, has been studied. These solvents, refluxing over powdered Bruceton coal, extract as a dispersion about 85 percent of the coal, corresponding to the total anthraxylon plus the translucent attritus coal fractions; they have, with the exception of α-naphthol, little dispersing action on Wyoming coal. α-Naphthol is unique in that it is a powerful solvent for both coals.

Little is known of the extraction mechanism of phenanthrene-type solvents except that there appears to be both a temperature specificity (only α- and β-naphthols show appreciable extracting action below 300° C.) and a structure specificity (anthracene, which is an isomer of phenanthrene, is a relatively much less efficient solvent, and its presence in a mixture with phenanthrene or carbazole strongly depresses the extracting power of the latter materials). Increasing the oxygen content of the coal by extensive heating in air results in a much lower degree of dispersion.

Knowledge of the extractability of coal by phenanthrene and other solvents is important to a study of the function of recycle oil vehicles used in the Bergius - I. G. process, in that a two-stage coal-hydrogenation process could be set up using extraction as a preliminary step to prepare a low-ash substrate that would decrease the difficulty of processing the "heavy-oil let-down" of the conventional process[74]

73/ Orchin, M., and Storch, H. H., Solvation and Hydrogenation of Coal: Ind. Eng. Chem., vol. 40, 1948, pp. 1385-1389.

74/ Savich, T. R., Pelipetz, M. G., Budy, W. A., Clark, E. L., and Storch, H. H., Stripping of Coal Hydrogenation Heavy-Oil Slurry: Ind. Eng. Chem., vol. 41, 1949, pp. 968-971.

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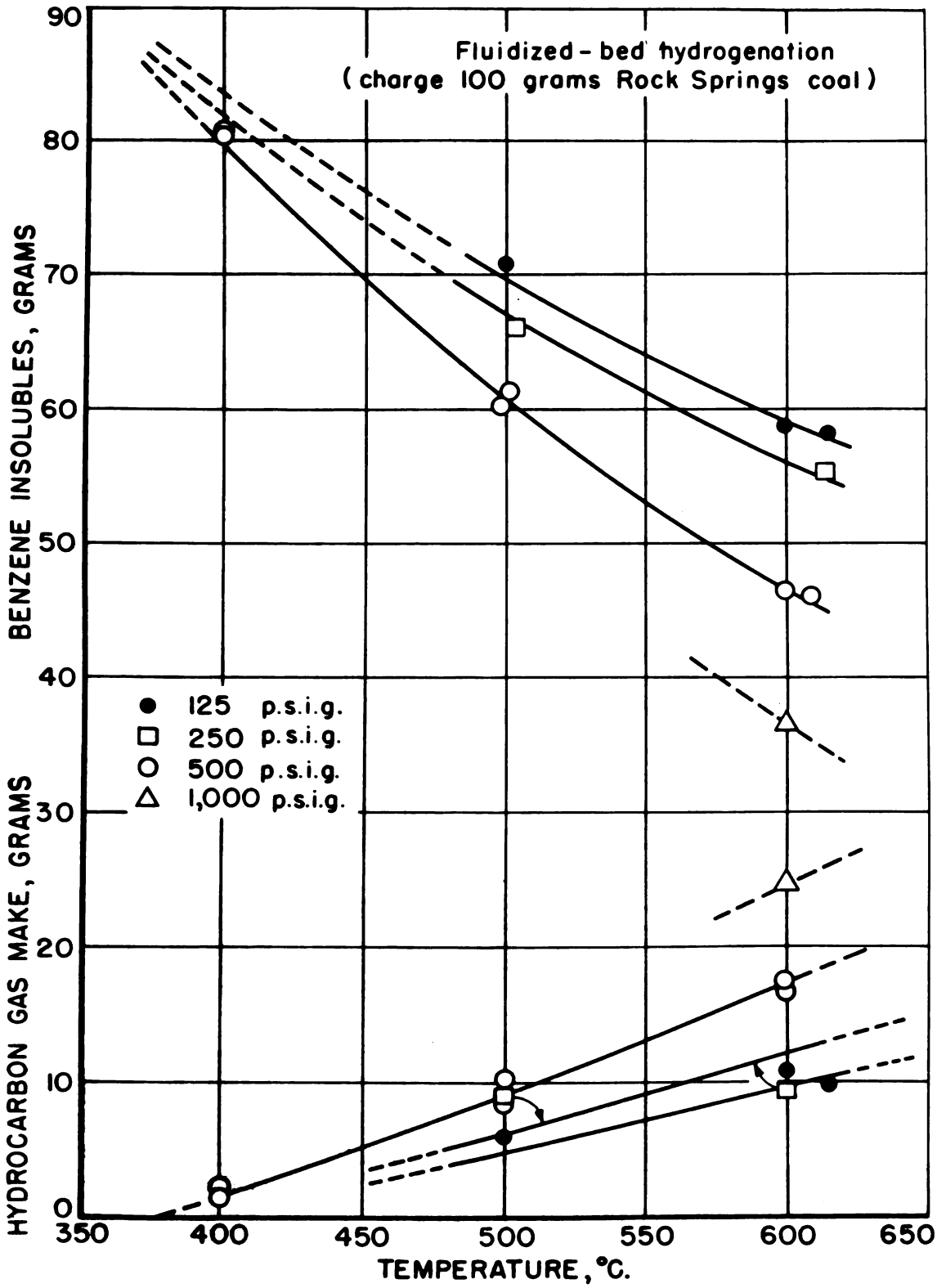

Figure 31. - Effect of pressure and temperature on benzene-insoluble residue and hydrocarbon gas production.

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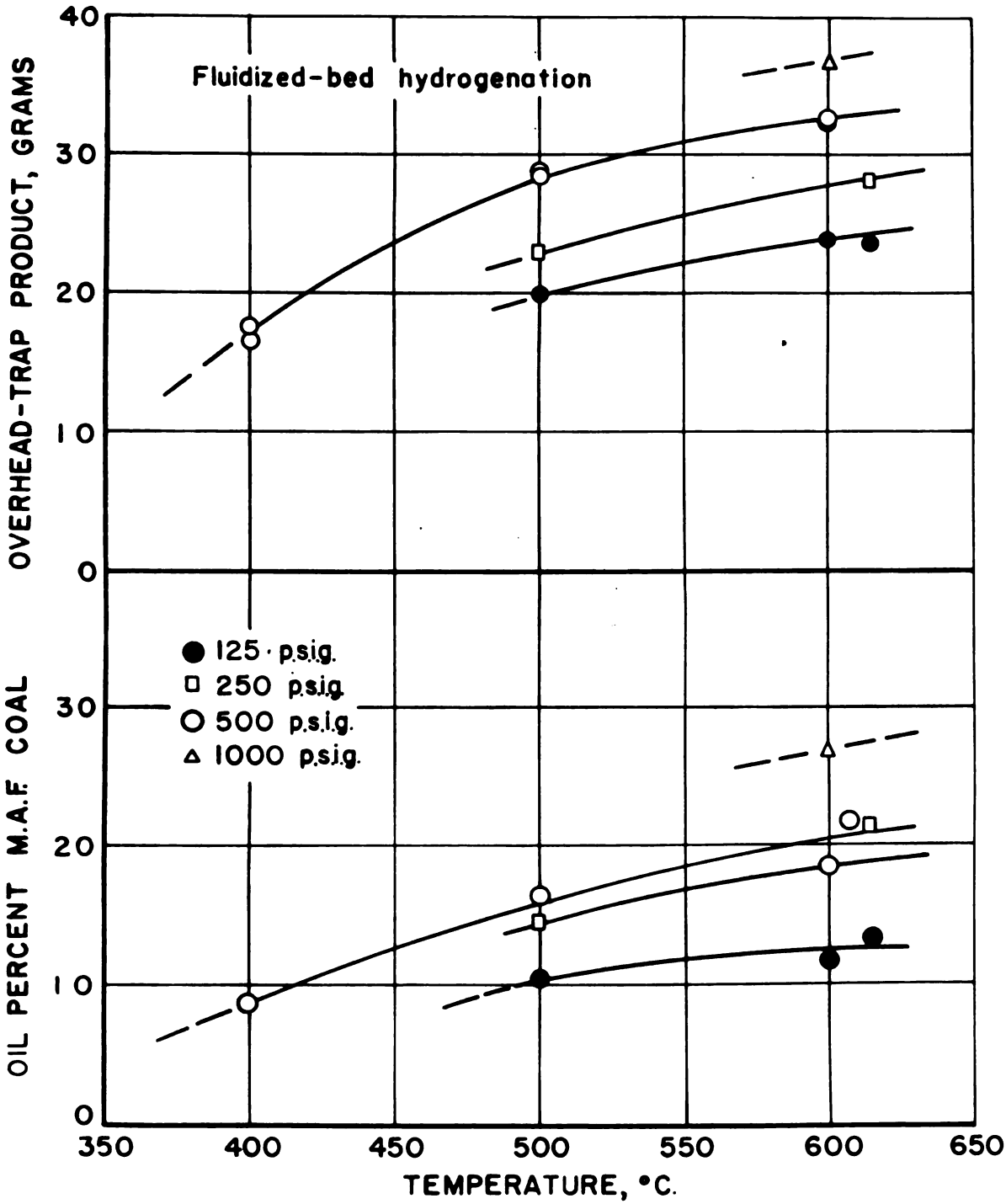

Figure 32. - Effect of pressure and temperature on overhead-trap product and oil production.

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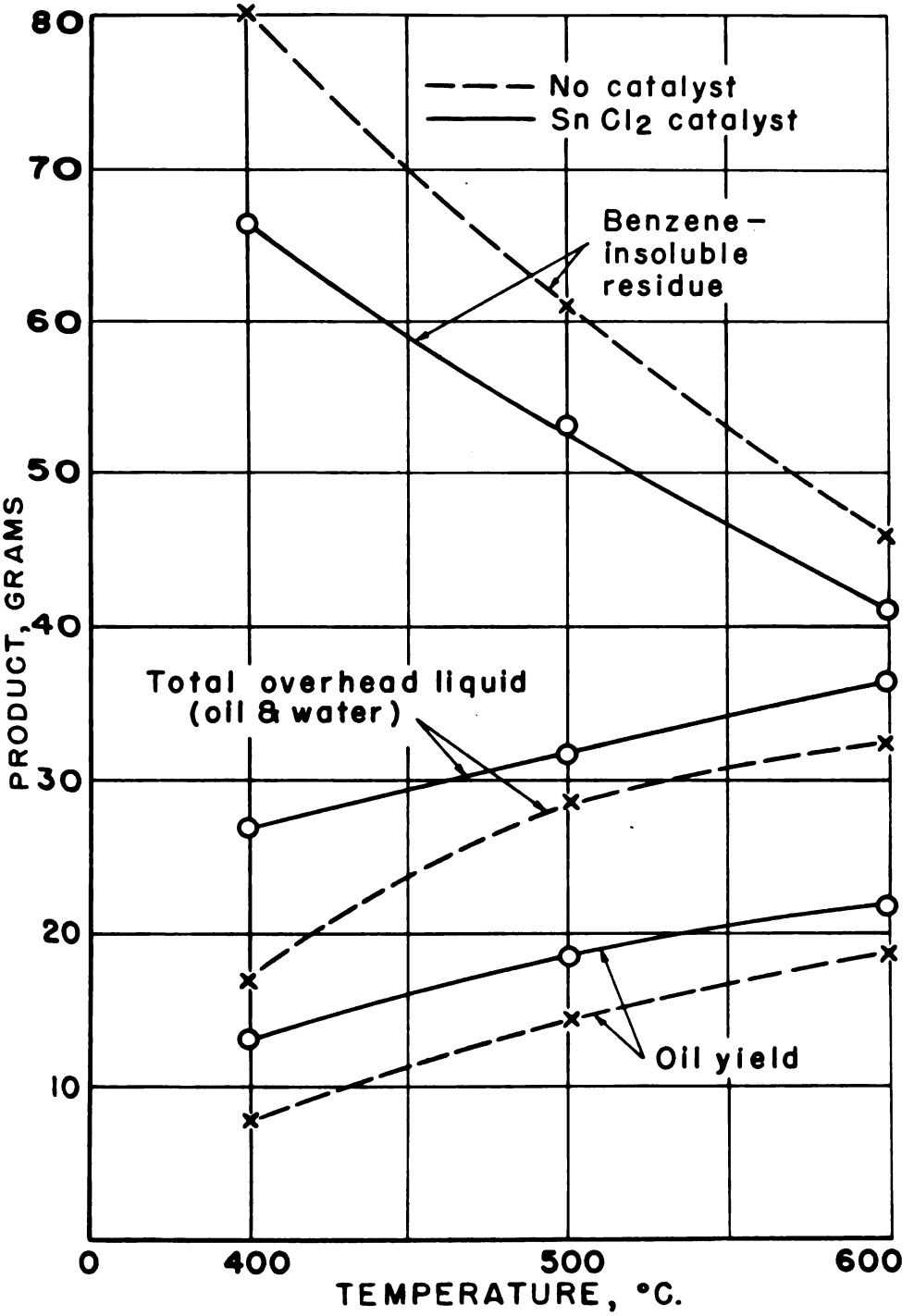

Figure 33. - Products of fluidized-bed hydrogenation in presence or absence of catalyst.

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or permit the hydrogenation to be carried out over a fixed-bed catalyst. A dispersion of Bruceton coal in phenanthrene has been shown to be as susceptible to hydrogenation as the original coal, and it was demonstrated that phenanthrene is not affected by the conditions and catalysts used for coal hydrogenation, thus permitting phenanthrene recovery. The substitution of phenanthrene by solvents obtained directly from coal is being investigated, and a coal-hydrogenation oil-product fraction boiling above 320° C. has been found to be as effective a solvent as phenanthrene.

Bench-Scale Process Development

Coal Hydrogenation in the Absence of Solvents

The hydrogenation of bituminous coal in the absence of vehicle is accomplished by suspending the coal particles, which are impregnated with catalyst in a stream of hydrogen in the reactor (fluidized bed). Such a process is of interest because of the considerable economies that may be achieved (1) by operating at a pressure of 50 to 70 atmospheres (in contrast to the 300 to 700 atmospheres employed in the conventional process) and (2) by utilizing the hydrogen that is available from the conversion of excess coal to coke in the reactor for the production of oil and gas.

Earlier studies[75] of the effects of contact time and temperature at initial hydrogen pressures of 1,000 to 10,000 pounds per square inch have been extended to include lower initial pressures (125 to 1,000 pounds per square inch) and higher temperatures (400°-600° C.) and indicate the lower limits for appreciable conversion to be 400 to 500 pounds per square inch and 500° C. Figures 31 and 32 show that operations can be so adjusted to convert from 15 to 27 percent of the moisture- and ash-free coal to oil and 12 to 27 percent to hydrocarbon gases. The omission of the usual catalyst (1 percent tin as $SnCl_2$ impregnated on the coal) results in decreased oil yields and increased benzene-insolubles as shown in figure 33. No advantages are to be gained by operation at high gas-flow rates. At decreased reaction times, however, although the production of oil is somewhat depressed, an improved utilization of hydrogen is obtained by virtue of the decrease in gasification that occurs simultaneously (see table 18).

TABLE 18. - Effect of reduction in reaction time on the hydrogenation of coal in a fluidized bed

(100 g. Rock Springs coal, 100- to 200-mesh, 500 p.s.i. initial H_2 pressure, reaction temp. = 600° C., rate of gas flow = 0.5 SCFM)

Test No.	Time at temp., hours	H_2 absorbed		Oil, percent m.a.f. coal	Asphalt, percent oil	Percent H_2 in oil	Percent O_2 in oil
		Percent m.a.f. coal	Percent oil				
70 ..	1	3.5	11.3	24.1	74	8.0	6.4
62 ..	.5	2.3	9.5	24.8	72	8.0	6.7
66 ..	a/0	1.6	7.8	22.1	68	8.0	6.5

a/ Heated to reaction temperature and cooling started immediately.

75/ Pelipetz, M. G., Kuhn, E. M., Friedman, S., and Storch, H. H., Effect of Catalysts on the Hydrogenolysis of Coal: Ind. Eng. Chem., vol. 40, 1948, pp. 1259-1264.

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Substitution of Water Gas for Hydrogen

It has been suggested that water gas might be substituted for the almost pure hydrogen now used industrially in the preparation of liquid fuels by the hydrogenation of coal. This would be an economic improvement, as the cost of compressed hydrogen constitutes about 50 percent of the total cost of liquid fuels prepared by the coal-hydrogenation process. Laboratory experiments at Bruceton have shown that it is indeed possible to replace hydrogen by water-gas under appropriate operating conditions. Because the water-gas shift reaction, viz., $CO + H_2O = CO_2 + H_2$, is quite fast under the conditions of coal hydrogenation, carbon monoxide is equivalent to hydrogen. However, no net decrease in cost is expected from this discovery because of the cost of the additional operation involved in removing carbon dioxide from the recycle hydrogen gas. The use of coke-oven gas as a source of hydrogen also is possible.76/

Another procedure for reducing the cost of hydrogen consists in using byproduct hydrocarbon gases. These may be obtained under pressures of 500 to 600 pounds per square inch, reacted with steam at that pressure, and the resulting mixture of hydrogen and carbon dioxide washed with water under pressure. In this way, the initial compression of hydrogen can be avoided, and, with a relatively small sacrifice in oil yield, enough byproduct hydrocarbons can be formed to supply all the hydrogen necessary for the process. An estimate of the reduction in cost of plant installation and operating cost to be expected from the use of byproduct hydrocarbon gas for hydrogen production is being made by the demonstration-plant staff.

Pilot-plant Operations

The installation of two pilot plants, one for the first or liquid-phase stage and one for catalyst testing in the second or vapor-phase stage has been completed. The liquid-phase plant has a capacity of about 10 gallons and the vapor-phase unit about 3 gallons of liquid product per day. Operation of both plants was begun in connection with tests on raw materials, catalysts, and procedures to be used in the larger demonstration plant at Louisiana, Mo.

Liquid-phase Hydrogenation

Operations in the liquid-phase hydrogenation pilot plant were begun in February 1949 and consisted of studies of a preliminary and orienting nature. During these operations, a pasting oil-catalyst mixture, a Rock Springs coal-pasting oil mixture, and Rock Springs coal-pasting oil-catalyst mixture were passed through the pilot plant under reaction conditions; minor changes in design were made as a result of these runs. The pilot-plant is now being operated with Rock Springs coal in a run for production of light oil.

Vapor-phase Hydrogenation

Figure 34 is a photograph of a high-pressure, fixed-bed, continuous catalytic unit that is being used to test catalysts for the demonstration plant at Louisiana, Mo., and to perform hydrogenation experiments on extracts or solutions of coal. Normal operating pressure for this unit is 10,800 pounds per square inch.

76/ Weller, S., Pelipetz, M. G., Kuhn, M., Friedman, S., and Clark, E. L., Hydrogenation of Coal in Batch Autoclaves with Coke-Oven Gas: Ind. Eng. Chem., vol. 41, 1949, pp. 972-973.

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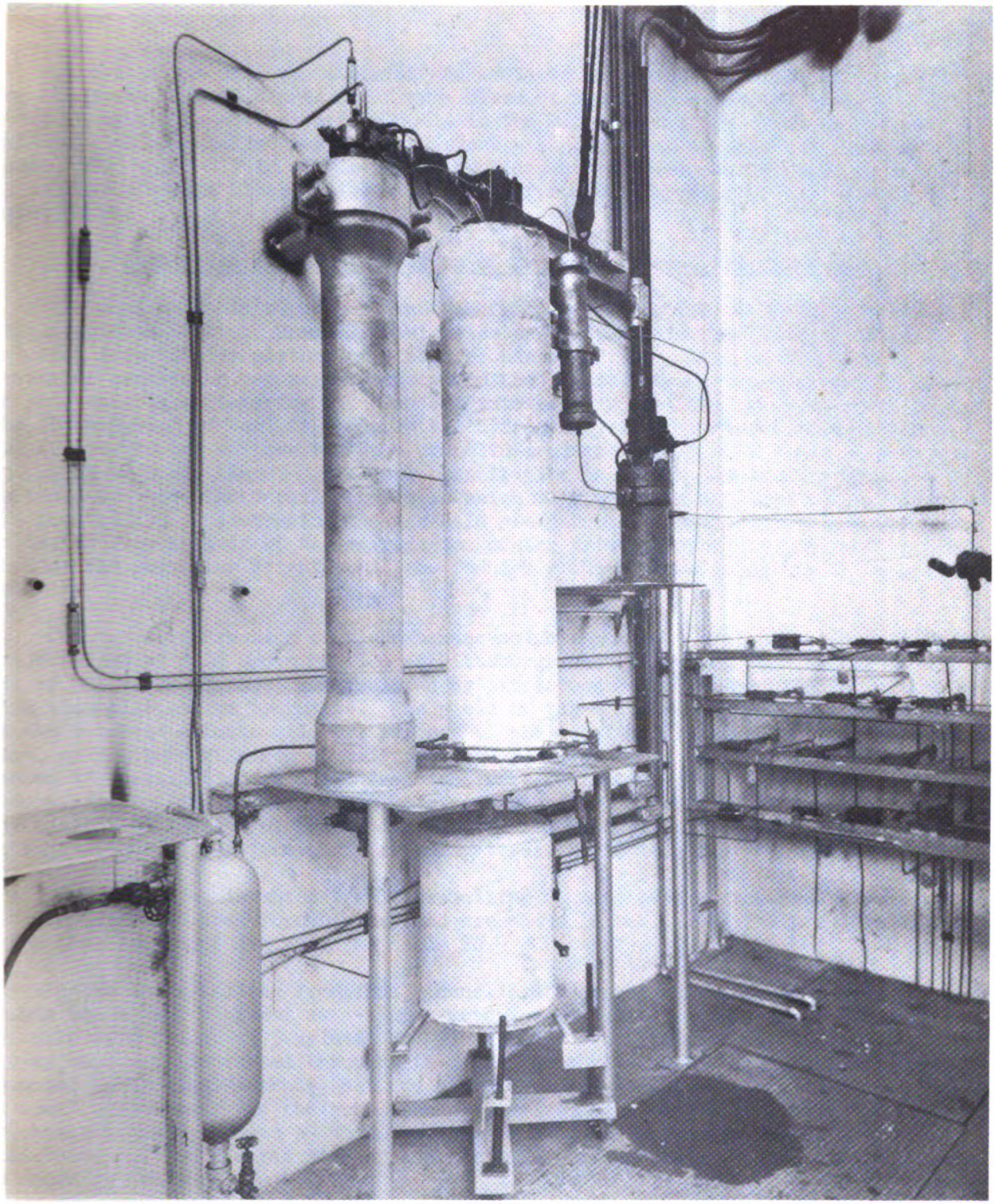

Figure 34. - Coal-hydrogenation vapor-phase catalyst testing unit.

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This unit was used to determine the activity of a German hydrogenation catalyst for the production of Diesel oil from a blend of light oils (boiling below 325° C.) obtained by hydrogenating various coals in the Pittsburgh pilot-plant operations. This particular catalyst was shown to produce a good aromatic gasoline (boiling point, 200° -325° C.) in single-pass operation, but several passes would be required to produce a usable Diesel oil that requires further saturation of the aromatic constituents.

Separation and Identification of Products of Coal Hydrogenation

The most valuable byproduct fraction from the hydrogenation of coal is the alkali-soluble portion, commonly designated as the tar-acid fraction.77/ This material is a complex mixture of acidic aromatic hydroxy compounds or phenols. Information concerning tar acids produced in the liquid-phase hydrogenation of coal is of considerable engineering and chemical interest, in that the critical reaction in the liquefaction of coal is probably the hydrogenolysis of carbon-to-oxygen bonds, and a study of the structure of the phenolic products should shed light on the constitution of coal. Knowledge of the constitution of these phenols also would serve to indicate those industrial markets in which they might be utilized as raw materials. The possibility of increasing the tar-acid yield from hydrogenation of coal can be estimated from a study of the degradation of the primary asphaltic products to learn whether the molecular weight could be reduced without the elimination of oxygen as water.

The tar-acid fraction of the n-hexane-soluble portion (oil) of the liquid product obtained by mild hydrogenation of Pittsburgh-bed (Bruceton) coal (to produce fuel oil as the major product) was isolated by extraction of a benzene solution of the oil with alcoholic sodium hydroxide. The yield of tar acids was 9.4 percent by weight of the n-hexane-soluble oil, 5.2 percent of the total oil produced in the hydrogenation, or 4.0 percent based on moisture- and ash-free coal. The tar acids were subjected to distillation, and 136 fractions were collected. On the basis of refractive indexes and boiling points, these were combined into 70 fractions. Chemical and physical investigation of selected fractions resulted in the isolation and identification, either as pure compounds or derivatives, of 16 individual phenols.

The quantities in which the lower-boiling (C_6-C_8) phenols occurred were determined by infrared spectrophotometry and are shown in table 19. The fractions in which they are found are shown in table 20.

In the lower-boiling fractions, phenol and o-cresol were isolated as pure compounds and m- and p-cresol as m- and p-methylphenoxyacetic acids. Four isomeric xylenols were found. 3,5-xylenol and 2,5-xylenol were isolated as pure compounds. 2,4-xylenol was identified through its N-(2-fluorenyl)-carbamate; 3,4-xylenol was not detected by chemical methods, but was shown by infrared spectroscopy to be present in small quantities. Of the ethyl-phenols, only one, m-ethylphenol, was identified by chemical means; 0- and p-ethylphenols were shown to be present by infrared spectroscopy. The distribution of the tar acids in the phenol-through-oxylenol range is shown in table 21.

77/ Columbic, N., Some Chemicals from Synthetic Liquid Fuels Processes: Bureau of Mines Rept. of Investigation 4467, 1949, 58 pp.

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TABLE 19. - Yields of tar acids from mild hydrogenation of Bruceton coal (to produce a fuel oil as the major product)

Compounds	Percent of hexane soluble oil	Percent of total oil	Pounds per ton of total oil	Pounds per tons of coal[1/]
Phenol	0.24	0.13	2.6	2.0
o-cresol	.37	.20	4.0	3.1
m-cresol	.55	.30	6.0	4.7
p-cresol	.25	.14	2.8	2.2
2,4-xylenol	.23	.13	2.6	2.0
2,5-xylenol	.14	.08	1.6	1.2
3,5-xylenol	.24	.14	2.8	2.2
3,4-xylenol	.05	.03	0.6	0.5
0-ethylphenol	.03	.02	0.4	0.3
m-ethylphenol	.42	.23	4.6	3.6
p-ethylphenol	.09	.05	1.0	0.8
3-methyl-5-ethylphenol ..	.27	.15	3.0	2.3
4-indanol	.19	.10	2.0	1.6
5-indanol	.13	.07	1.4	1.1
Total	3.20	1.77	35.4	27.6

1/ Dry, ash-free coal.

TABLE 20. - Fractions in which identified phenols were found

Fraction	B.P., °C.	Press., mm. Hg	Compounds	Identification
4-7	97-99	40	Phenol	Pure and as phenyl N-phenylcarbamate.
8-21	99-102	40	o-cresol	Cineole derivative.
			Phenol	As tribromophenol.
22-24	100-104	40	o-cresol	Pure and as N-phenylcarbamate derivative.
34-40	114-115	40	m-cresol	As tribromo m-cresol.
			p-cresol	As aryloxyacetic acid derivative.
44-47	116-120	40	2,5-xylenol	Pure and as N-phenolcarbamate derivative.
			2,4-xylenol	As N-(2-fluorenyl) carbamate derivative.
48-51	121-125	40	2,4-xylenol	p-Nitrobenzylether derivative.
			2,3-xylenol (?)	Aryloxyacetic acid derivative (but not by infrared).
			Mesitol (?)	Methoxytrimesic acid - trimethyl ester (but not by infrared).
56-61	129-130	40	M-ethylphenol	Aryloxyacetic acid derivative.
			o-ethylphenol	Infrared spectrometry.
			p-ethylphenol	Infrared spectrometry.
63-65	130-131	40	3,5-xylenol	Pure as N-phenylcarbamate derivative.

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TABLE 20. - Fractions in which identified phenols were found (Cont'd.)

Fraction	B.P., °C.	Press., mm. Hg	Compounds	Identification
77-82	141-145	40	3-methyl-5-ethylphenol	As benzoate, n-phenylcarbamate and aryloxyacetic acid derivatives, infrared spectrometry.
85	149-151	40	4-indanol	Pure and ultraviolet analysis.
99	156-159	40	5-indanol	Pure and ultraviolet analysis.
105-107	161-164	40	Symmetrical C_{10} phenols and methyl homologues of 5-indanols.	Spectroscopic data and chemical derivatives.
117-118	165-168	30	o-phenylphenol	Pure and as aryloxyacetic acid derivative.
135	197-203	30	p-phenylphenol	Pure and ultraviolet analysis.

TABLE 21. - Distribution of the tar acid in phenol-through-oxylenol range

Compounds	Grams in tar acid fractions	Percent of total tar acids	Percent of total C_6-C_8 fractions
Phenol	111.5	2.6	9.3
o-cresol	169.9	3.9	14.1
m-cresol	255.7	5.9	21.3
p-cresol	115.3	2.7	9.6
2,4-xylenol	103.2	2.4	8.6
2,5-xylenol	63.7	1.5	5.3
3,5-xylenol	115.9	2.7	9.6
3,4-xylenol	20.6	0.5	1.7
o-ethylphenol	14.9	0.3	1.2
m-ethylphenol	188.0	4.4	15.6
p-ethylphenol	43.8	1.0	3.6
	1,202.5	27.9	99.9

A new analysis for hydroxyl oxygen in coal-hydrogenation oils was developed, which involves reaction of the oil in dry pyridine with Grignard reagents; hydroxylic compounds are known to enter quantitatively into this reaction with the formation of stoichiometric amounts of methane. Previous analyses were based on an extraction of the oil with aqueous or alcoholic alkali. Aqueous alkali is not suitable extraction agent because of the limited solubility of high-boiling phenols in such a medium. The use of alcoholic alkali improves the extractibility but involves the troublesome question of distribution of neutral oil in the system. Compounds other than the hydroxylic type, containing active hydrogen atoms, would react with Grignard reagents and interfere with the determination of oxygen. The principal source of such an error is primary amines, but investigation has shown that the heavier oils obtained from coal hydrogenation contain little amino nitrogen; most of the nitrogen present is cyclic in nature, as indicated by the fact that it is eliminated only slowly from coal. This method cannot, of course, discriminate between phenolic and aliphatic hydroxyl groups, but treatment with Grignard reagents of a benzene extract of an alkali extract of the

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oil showed that approximately 29 percent of the total oxygen in the oil was phenolic in nature.

Ultraviolet spectrometry continues to be of great importance in the aromatic cyclodehydrogenation studies[78] [79] that are being made to assist in the identification of polynuclear hydrocarbons present in coal hydrogenation oils and to provide an understanding of their mode of formation.

The preparation of isomeric 1-aminofluorene was reported.[80] The synthesis consisted essentially of the oxidation of fluoranthane to fluorenone-1-carboxylic acid, reduction of the latter to fluorene-1-carboxylic acid, and replacement of the carboxyl group by an Amino group.

A new general synthesis of fluoranthene and its derivatives was reported.[81] The anhydride resulting from the condensation of fluorene and maleic anhydride was cyclized to 1-carboxy-3-keto-1,2,3,10 b-tetrahydro fluoranthene, which was then reduced and dehydrogenerated to fluoranthene-1-carboxylic acid. The carboxyl group was esterified, and Curtius degradation of the ester gave 1-aminofluoranthene.

Installation of batch distillation equipment has been completed. Batches of 50 to 500 gallons of oil can be distilled in this equipment to provide large samples of gasoline, Diesel oil, or byproduct tar acids for practical tests and for precision separations to be conducted by the distillation group of the organic chemistry section. The latter has been engaged in development of standards for vacuum distillation and in design and erection of a number of precision stills. A photograph of the pilot-plant scale equipment is shown in figure 35, and of a typical still assembly in figure 36.

LIQUEFACTION OF COAL BY HYDROGENATION OF CARBON MONOXIDE: RESEARCH AND DEVELOPMENT

The process for the production of liquid fuels by the hydrogenation of carbon monoxide in the internally cooled (by oil circulation through a bed of catalyst granules) converter has been greatly improved, and relatively cheap, durable catalysts have been found for the process. Since 1942, industrial activity in the United States has centered about the application of the fluidized catalyst technique to the synthesis. The Bureau of Mines, however, has continued the development of other types of Fischer-Tropsch processes, and a second process is now in the pilot-plant stage of development. In this process, the finely powdered catalyst is suspended in a high-boiling oil, and the synthesis gas is bubbled through the suspension. Rate measurements in laboratory reactors and X-ray and magnetic studies of catalysts have resulted in a clearer understanding of the nature of the catalytic processes involved.

78/ Woolfolk, E. O., and Orchin, M., Preparation of 2,4,7-Trinitro fluorenone: Org. Syn., vol. 28, 1948, p. 91.

79/ Orchin, M., Woolfolk, E. O., and Reggel, L., Aromatic Cyclohydrogenation VIII. Experiments with 2-Benzoyl - 5,6,7,8-tetrahydronapthalene; Jour. Am. Chem. Soc., vol. 71, 1949, p. 1126.

80/ Bergman, E., and Orchin, Milton, Synthesis of 1-Aminofluorene: Jour. Am. Chem. Soc., vol. 71, 1949, pp. 1111-1112.

81/ Bergman, E., and Orchin, Milton, Synthesis of Fluoranthene and Its Derivatives: Jour. Am. Chem. Soc., vol. 71, 1949, pp. 1917-1918.

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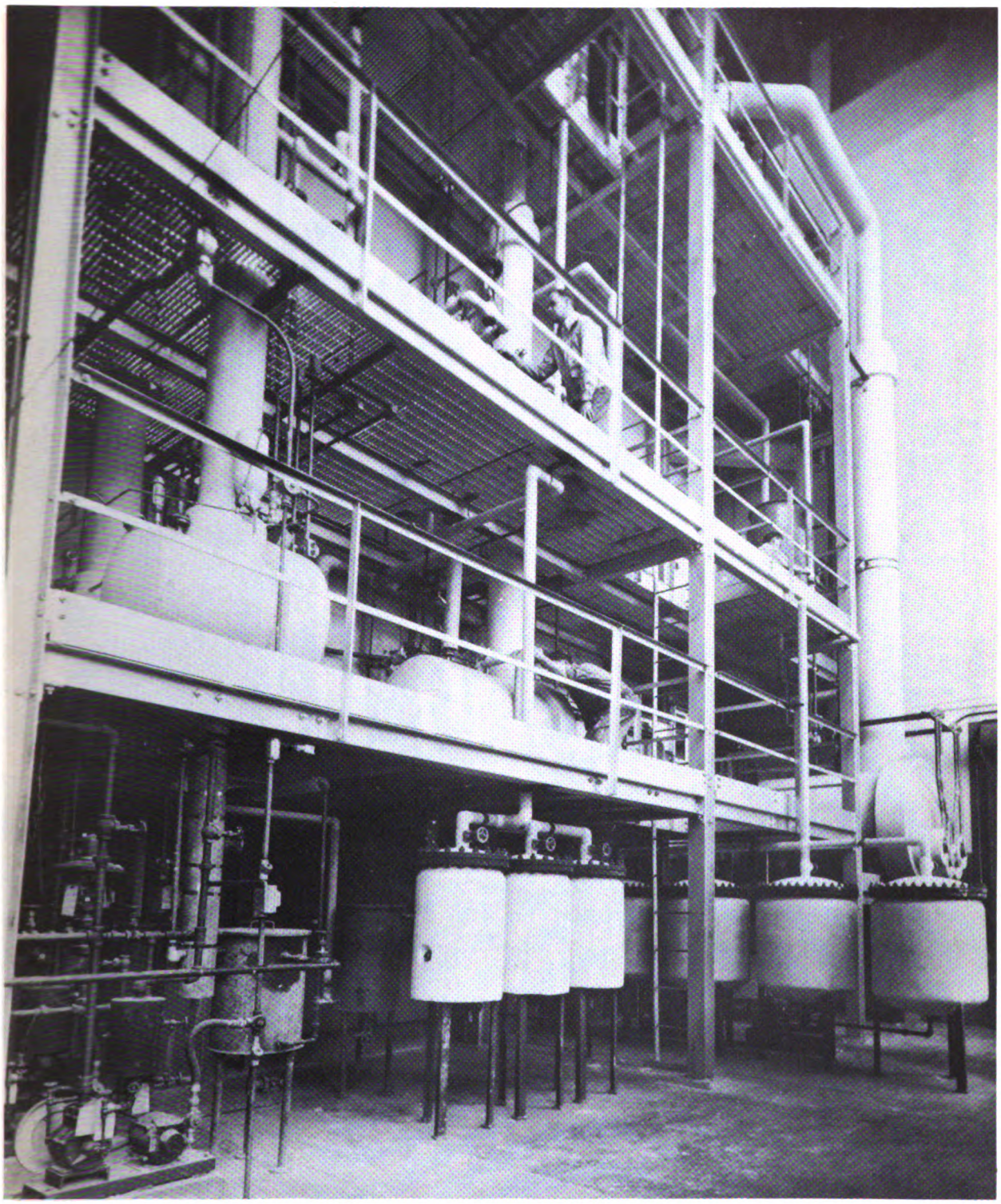

Figure 35. - 100- to 500-gallon batch-distillation equipment.

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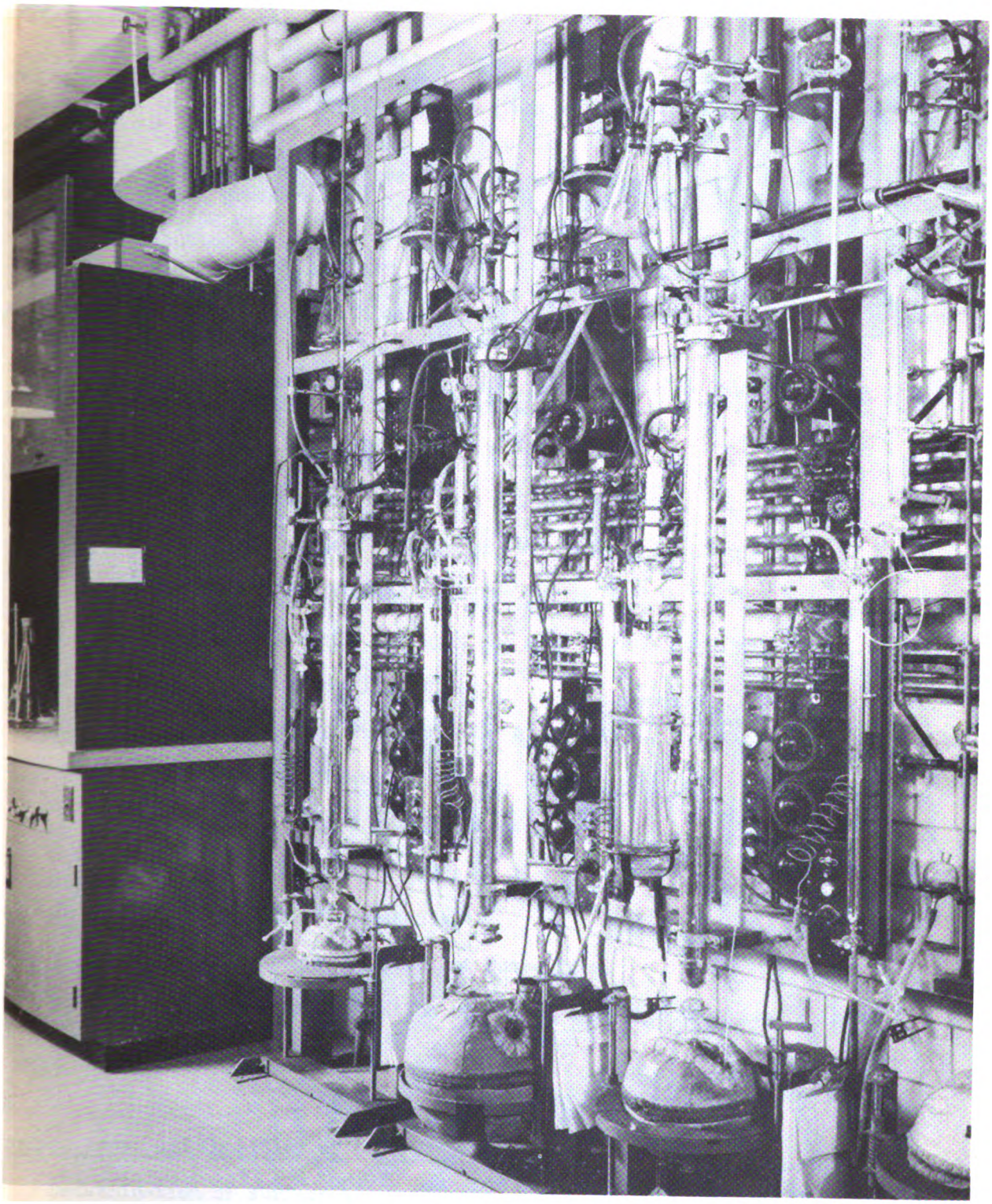

Figure 36. - Typical distillation assembly.

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Cost estimates on Fisher-Tropsch processes, starting with coal as the source of carbon, show that a very large fraction of the cost of production of liquid fuels is the cost of synthesis-gas production. A study of this phase of the process, therefore, is of obvious importance.

The dependence of the Fischer-Tropsch synthesis upon space velocity of synthesis gas, temperature, pressure, and synthesis-gas composition was reviewed.82/ The effects of these operating variables upon the over-all synthesis rate and upon the molecular weight distribution and degree of unsaturation of synthesis products were interpreted in terms of a simplified, unitary picture of the elementary processes occurring during synthesis. Recent work on the role of metal carbides as intermediates in the synthesis was considered. With cobalt catalysts, bulk carbide seems to be neither an intermediate nor a catalyst for the synthesis.

The research and development program for synthesis of liquid fuels by hydrogenation of carbon monoxide is outlined as follows:

A. Reaction Mechanism Studies.

   1. Surface Areas and Densities of Iron Catalysts.

   2. Effect of Carriers and Promoters in Cobalt Catalysts and Role of Bulk Metal Carbides in the Synthesis.

   3. Effect of Chloride Ion upon the Activity of Iron Catalysts.

B. Catalyst Activity and Durability.

C. Study of Fundamental Properties of Fixed and Fluidized Beds.

   1. Heat Transfer Through Fluidized Beds.

   2. Flow Through Fixed Granular Beds.

   3. Flow Through Fluidized Beds.

D. Process Development, Bench Scale.

E. Process Development, Pilot-plant Operations.

F. Separation and Identification of Products of Hydrogenation of Carbon Monoxide.

Reaction Mechanism Studies

Surface Areas and Densities of Iron Catalysts

Determination of surface area and mercury and helium densities of fused iron catalysts showed that, during reduction, the external volume of the catalyst particle

82/ Weller, Sol , Effect of Operating Variables on the Fischer-Tropsch Synthesis: Bureau of Mines Rept. of Investigation 4405, 1949, 8 pp.

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remains the same, and pores are developed by removing oxygen from the interior of the grain. The average pore diameters (330 to 350$\mathring{A}$) remain very nearly constant throughout the reduction and are not widely different from those found in reduced cobalt catalysts (327 to 672$\mathring{A}$). Surface areas were found to vary almost linearly with extent of reduction. At 90 to 100 percent reduction, however, it appears that some sintering occurs, as the surface area does not increase as much as the pore volume.

Similar studies with precipitated iron catalysts suggest an explanation for the greater activity of precipitated catalysts as compared to fused catalysts. The surface of the precipitated catalysts may be more accessible to the reacting gases owing to the presence of pores with diameters about three times as large as those found in fused catalysts. Similarly, reduced cemented catalysts, having pores about 1,400$\mathring{A}$ in diameter, are more active than reduced fused catalysts (pore diameter, 300$\mathring{A}$).

Effect of Carriers and Promoters in Cobalt Catalysts and Role of Bulk Metal Carbides in the Synthesis

A number of commercially available American kieselguhrs were studied in connection with promoted cobalt catalysts.83/ Acid-extracted natural kieselguhrs produced catalysts of the highest activity. The density of the catalyst varied directly with the density of the kieselguhr, and the distribution of products changed with density of the catalysts, the denser catalysts forming a greater percentage of light hydrocarbons and carbon dioxide. Kieselguhr as a carrier was primarily effective in preventing a decrease in bulk volume of the catalyst on reduction. The promoters were found to prevent excessive decreases in surface area on reduction, and carbon monoxide chemisorption studies showed that an appreciable fraction of the surface was occupied by promoter.84/ Decrease in the accessibility of the surface of these catalysts, either by deposition of excessive quantities of wax or by reduction of average pore diameter by pelleting, caused an increase in the fraction of light hydrocarbons and, in some cases, a decrease in activity.85/

X-ray diffraction patterns of cobalt and iron carbides combined with the magneto-chemical constants and earlier determinations of such patterns and constants for the iron oxide phases86/ have provided the essential elements for analysis of Fischer-Tropsch catalysts by physical methods. Although little or no metal carbide

83/ Anderson, R. B., Krieg, A., Seligman, B., and Tarn, W. H.: Fischer-Tropsch Cobalt Catalysts. Influence of Type of Kieselguhr: Ind. Eng. Chem., vol. 40, 1948, pp. 2347-2351.

84/ Anderson, R. B., Hall, W. K., and Hofer, L. J. E.: Studies of the Fischer-Tropsch Synthesis. IV. Properties of Reduced Cobalt Catalysts: Jour. Am. Chem. Soc., vol. 70, 1948, pp. 2465-2472.

85/ Anderson, R. B., Hall, W. K., Krieg, A., and Seligman, B., Studies of the Fischer-Tropsch Synthesis, V. Activities and Surface Areas of Reduced and Carburized Cobalt Catalysts: Jour. Am. Chem. Soc., vol. 71, 1949, pp. 183-188.

86/ Storch, H. H., Anderson, R. B., Hofer, L. J. E., Hawk, C. O., Anderson, H. C., and Golumbic, N.: Synthetic Liquid Fuels from Hydrogenation of Carbon Monoxide. Part I. Review of Literature; Bureau of Mines Research on Effect of Catalyst Preparation, Reduction, and Induction Procedures on Activity; Correlation of Physical Properties of the Catalysts with Their Activity: Bureau of Mines Tech. Paper 709, 1948, 213 pp.

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is formed when the synthesis is conducted on cobalt catalysts[87] and extensive precarbiding greatly reduces the activity of these catalysts, the reverse is true for iron catalysts. Precarbiding of iron catalysts prepared by precipitation from solutions of iron and promoter metal nitrates yields very active catalysts. Where no precarbiding is done, iron catalysts form large amounts of iron carbide during the first few weeks of operation. Thermodynamically, however, it is impossible to account for more than about 0.005 percent of the total olefins in the product of the synthesis from hydrogen on iron carbide, and no appreciable amounts of saturated hydrocarbons larger than hexane are possible. It is probable, therefore, that the beneficial action of iron carbides is confined to that of a promoter.

The relationship between the carbides of formula Fe_2C, which were reported in thermomagnetic studies of Pichler and Merkel, and those reported in X-ray diffraction studies of various workers such as Hägg, Jack, and Halle and Herbst was established. The existence of two carbides of iron with the formula Fe_2C was confirmed. One carbide is identical with that described by Hägg; its Curie point is 247° C. The other carbide is identical with the hexagonal close-packed described by Halle and Herbst; its Curie point is 380° C. The latter carbide is converted above 300° C. to the Hägg carbide, which in turn is decomposed at 550° C. to cementite, Fe_3C, and carbon.[88]

After reduction and carburization of precipitated iron catalyst, X-ray diffraction analysis showed the presence of hexagonal, close-packed Fe_2C. The mode of transition from Fe_2C-Hägg to cementite, Fe_3C, was studied by following the course of the magnetic curves between the Curie points of the carbides; the transition was found to be discontinuous, which means that precipitation starts with the formation of nuclei of the new crystal habit, followed by their growth at the expense of the existing structure.

The change in the composition of a fused iron catalyst was studied during its use in the medium-pressure Fischer-Tropsch synthesis. X-ray diffraction and thermomagnetic analyses showed that Hägg carbide (Fe_2C) was formed very rapidly during the beginning of the experiment; a maximum amount had been formed sometime during the first three days of operation, after which it decreased linearly with time. No carbides other than Fe_2C-Hägg were identified. Similar examination of used iron catalysts after catalyst-testing studies showed that, of the three known ferromagnetic iron carbides, the hexagonal, close-packed Fe_2C is rarely formed with fused catalyst under the usual synthesis conditions, and that cementite, Fe_3C, does not appear to be formed during the synthesis except by over-heating and simultaneous decomposition of the other forms of carbide.

Because of some uncertainty in the conclusions from the work with radioactive carbon as a tracer, kinetic and isotopic tracer studies with nonradioactive C^{13} isotope were begun. An apparatus was constructed for this work in which the reaction rate is measured in terms of volume changes by means of a gas burette as the gases are circulated over the catalyst samples at constant temperature and pressure. The first study involved the rate of exchange of carbon between carbon monoxide and a fully carbided cobalt catalyst. As this catalyst contained a small amount of

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87/ Hofer, L. J. E., Cohn, E. M., and Peebles, W. C., Isothermal Decomposition of the Carbide in a Carburized Cobalt Fischer-Tropsch Catalyst: Phys. and Colloid Chem., vol. 53, 1949, pp. 661-669.

88/ Hofer, L. J. E., Cohn, E. M., and Peebles, W. C., The Modifications of the Carbide Fe_2C; Their Properties and Identifications: Jour. Am. Chem. Soc., vol. 71, 1949, pp. 189-195.

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graphite as a pelleting lubricant, the rate of exchange between carbon monoxide and graphite was investigated as a preliminary step. Enriched carbon monoxide, containing 3.4 percent $C^{13}O$ and prepared as described in last year's report, was circulated over the graphite at 200° C. and 306 mm. mercury pressure for 17.5 hours. The enrichment of the carbon monoxide before and after circulation was determined in the mass spectrometer. No loss in enrichment was detected. The exchange between carbon monoxide and graphite is therefore of no significance in rate studies on this catalyst.

One of the worst features of Fischer-Tropsch catalysts is their tendency to form relatively inert free carbon during the synthesis. This carbon plugs up the reaction vessel in fixed-bed converters; in fluidized-bed converters it exerts a disintegrating effect on the catalyst particles, converting them into a fine dust that cannot be retained in the converter. In addition, it is thought that carbon deposition exerts a specific poisoning action on the Fischer-Tropsch reaction. Carbon may very probably be formed by way of carbide, as follows:

$$2CO + xM \text{ ..... } M_xC + CO_2 \qquad (1)$$

$$M_xC \text{ ..... } xM + C \text{ (free)} \qquad (2)$$

This theory is supported by the fact that only those metals that form metastable carbides are known to form free carbon from carbon monoxide. Reaction (2) is simply the thermal decomposition of the carbide involved.

As subject for a study to determine the nature of this decomposition reaction, nickel carbide was chosen, because (1) it could be prepared in a well-crystallized and pure form, (2) its crystal structure is well known, and (3) it is, like carburized cobalt catalyst, nonferromagnetic. Therefore, the thermal stability of the carbide (weight ratio, C:Ni = 0.0722:1) could be studied magnetically by the same method used previously for Co_2C, and the appearance of ferromagnetism could be ascribed to the formation of ferromagnetic nickel metal, a measure of the process of the decomposition. Results of the study showed that the apparent energy of activation (67 kg. cal./mol) for the decomposition of nickel carbide is of the same order of magnitude as that found for the decomposition of a carburized cobalt catalyst (54.3 kg. cal./mol). The decomposition of nickel carbide was found to be preceded by an induction period that decreased with increasing temperature. This induction period indicates that the reaction requires the presence of nuclei that are difficult to form, but which, when formed, allow the reaction to proceed at a relatively rapid rate. Inhibiting the formation of these nuclei by various additives would, theoretically, delay the thermal decomposition.

Effect of Chloride Ion upon the Activity of Iron Catalysts

Early in the development of the Fischer-Tropsch catalysts, it was found that active catalysts could not be prepared from ferric chloride. However, precipitated catalyst of composition $75Fe^{+2}$-$25Fe^{+3}$-20Cu-0.2K_2CO_3, which was prepared from ferrous, ferric, and cupric chlorides according to the procedure of Pichler, produced an average yield of 88.3 g. of C_5+ hydrocarbons per cubic meter of water gas when tested for 46 days at 7 atmospheres pressure and an average temperature of 229° C. Tests made with several preparations in which the constituents of this catalyst were varied showed that the ferrous iron was the significant constituent.

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Chemical analysis (table 22) showed that the chloride content of the inactive ferric-iron catalysts was at least 20 times greater than that of the active ferrous-ferric preparations. On the basis of this information and additional data obtained by X-ray diffraction analysis, the following explanation of the deactivating effect of chloride is proposed.

TABLE 22. - Effect of presence of chloride in precipitated iron catalysts upon their activity in the Fischer-Tropsch synthesis (SHV = 100, pressure = 7 atmospheres)

Catalyst No.	Composition	Synthesis temperature, °C.	Contraction, percent	Average C_{5+} hydrocarbon yield, g./m3a/	Chloride, percent of raw catalyst
L3004	75^{+2}-$25Fe^{+3}$-20Cu-$0.2K_2CO_3$	229	66	88.3	0.04
L3008 b/	$100Fe^{+2}$-20Cu-$.5K_2CO_3$ ..	239	53	75.8	.02
L2001	$100(Fe^{+2}+Fe^{+3})$-$0.2K_2CO_3$	238	64	73.8	c/
L2003	$100Fe^{+3}$-$0.2K_2CO_3$	263	28	16.0	.56
L3006	$100Fe^{+3}$-10Cu-$0.2K_2CO_3$..	282	37	12.7	.92

a/ G./m³ of feed gas.
b/ Catalyst disintegrated.
c/ Precipitated from nitrates.

1. In the presence of chloride and ferric iron alone, β $Fe_2O_3.H_2O$ was produced, which retained chloride in the crystal structure. During synthesis, chloride present on active centers deactivated the catalyst.

2. In the presence of chloride and ferrous and ferric iron (at least 35 percent Fe^{+2}) or ferrous iron alone, the formation of β $Fe_2O_3.H_2O$ was inhibited. Instead, magnetite, Fe_3O_4, or ferrous hydroxide, $Fe(OH)_2$, or a mixture of both, was formed; from this crystal structure, chloride was easily removed by washing.

Catalyst Activity and Durability

The mechanical stability of new catalysts and the control of wax concentration in the recirculated oil are being investigated in the laboratory in connection with the internally cooled, oil-circulation process. Progress is being made towards an early solution of the problem of very cheap, durable, and adequately active catalysts for the internally cooled process. Because of the relatively expensive and difficultly reproducible nature of the fused, synthetic-ammonia type of iron catalyst, the use of readily available iron ores and industrial byproduct iron oxides is being studied. Starting with either finely divided magnetite ore concentrated by magnetic separation, or with mill scale from the steel industry, active and durable catalysts were prepared. The preparation consisted in making a thick paste of the finely powdered iron oxide with an aqueous solution of the nitrates of promoters such as aluminum, potassium, magnesium, and calcium, extruding the paste into small rods about 3/16 inch by 1/2 inch, drying, sintering (local fusing) at 500° to 600° C.,

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crushing to 8- to 10-mesh, and reducing with hydrogen. Catalysts so prepared were found to be much more active than the fused synthetic-ammonia type. They were not, however, sufficiently resistant to mechanical disintegration by the hydraulic action of the circulating oil in the internally cooled reactor. Improved mechanical stability is being sought by the addition of binders such as clay, silica gel, etc. The cost of these catalysts will not exceed about 5 cents per pound. Alkali-impregnated glomerules made from iron-oxide powder also appear to merit further investigation. The wet iron-oxide powder is compacted with a high-frequency vibrator, rolled into rough spheres, sintered at 1,200° C., and impregnated with alkali. After reduction at 450° C. for 20 hours, the glomerules were operated in the synthesis at a temperature of 235° C. In general, the production of C_{4+} hydrocarbons increases with the alkali content of the catalyst up to about 2.1 parts of K_2O per 100 parts of Fe. A fused iron catalyst containing 2.76 K_2O per 100 Fe was found to be relatively inactive (operating temperature about 300° C.). With cemented catalysts, the optimum concentration of K_2O appears to lie between 0.5 and 1.0 parts per 100 Fe.

Magnetite powder cemented with 5 percent of borax, heated to 800° C. for 10 minutes, followed by reduction at 450° C. for 20 hours, was operated in the synthesis at 250° C. This type of catalyst has hitherto been described as "sintered" instead of "cemented", but in the patent literature the term "sintered" is usually applied to catalysts in which the active component has been modified drastically by heat. In the present catalyst, the particles of iron oxide are cemented into granules by the borax, and no such drastic change is considered as taking place.

Work has continued on the duplication of the German synol catalyst ($Fe-Al_2O_3-CaO-K_2O-SiO_2$), which was found to be much more active than the usual synthetic-ammonia type catalysts, comparing favorably in activity and product distribution with the best precipitated catalysts. Previous attempts to duplicate the synol catalyst have been unsuccessful. Recently, however, information was found in captured German documents indicating that the best German synol catalyst was made by re-fusing an ammonia-type catalyst with carbonyl iron. The only significant differences between the preparation and composition of the synol catalyst and those of the usual synthetic-ammonia type catalysts are that (a) the synol catalyst contains calcium oxide in addition to the usual promoters; (b) the synol catalyst was remelted, and carbonyl iron was added to the melt; and (c) the synol catalyst was in the form of a fine powder. Comparison of the synol catalyst with synthetic-ammonia type catalysts of various particle sizes and containing as much as 30 percent carbonyl iron has demonstrated that (b) and (c) can explain most of the greater activity of the synol catalyst. Thus, the activity of a fused catalyst appears to vary inversely with the particle diameter, and it appears that accessibility of the catalyst surface is an important factor in the Fischer-Tropsch synthesis. This conclusion is supported by the results of mercury- and helium-density measurements.

Study of Fundamental Properties of Fixed and Fluidized Beds

Heat Transfer Through Fluidized Beds

A study of the physics of heat transfer through fluidized beds of round and sharp silica sands and iron Fischer-Tropsch catalysts was completed.89/

89/ Leva, M., Grummer, M., Weintraub, M., and Storch, H. H., A Study of Fluidization of an Iron Fischer-Tropsch Catalyst: Chem. Eng. Progress, vol. 44, 1948, pp. 707-716.

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Flow Through Fixed Granular Beds

A monograph has been completed for the calculation of pressure drop across fixed particle beds for viscous fluid flow; similarly, a correction chart has been prepared for the transitional range where viscous flow changes to turbulent flow. A general pressure-drop formula has been found to be valid for the laminar, transitional, and turbulent regions of fluid flow through fixed beds of any particle size. This work was done in connection with the accurate estimation of the energy requirements of the Fischer-Tropsch hot-gas-recycle process and other processes in which large linear velocities of gases through packed tubes are necessary.90/ Equations were derived that permit the evaluation of shape factors for ring or cylindrical packings.91/

Flow Through Fluidized Beds

The fundamental characteristics of fluidized systems were studied.92/ The fluidization of anthracite coal was shown to follow the previously established correlation for silica sands and iron Fischer-Tropsch catalyst.93/ 94/ The phenomenon of channeling during a fluidization run, which occurs after bed expansion and prior to minimum fluidization of the particle bed, has been expressed in terms of a "channeling factor." It was observed that the channeling factor decreases as the average particle diameter increases; it increases as the irregularity and density of the particle increases.

Process Development, Bench Scale

Cost estimates on Fischer-Tropsch processes, starting with coal as the source of carbon, show that a very large fraction of the cost of production of liquid fuels is the cost of synthesis-gas production and purification. It is apparent, therefore, that for industrial purposes such processes must use the synthesis gas in a very efficient manner; that is, only a minimum of gaseous hydrocarbons should be produced, and the usage ratio of hydrogen to carbon monoxide should be adjusted by selection of catalyst and operating conditions so that it is identical with the ratio in the cheapest synthesis gas.

A study of hydrogen-to-carbon monoxide usage ratios for various types of iron catalysts showed that, in the case of fused synthetic-ammonia-type catalysts, wide variations of catalyst composition and synthesis temperature caused only small changes in the usage ratio, whether the catalysts were promoted or unpromoted. Cemented and sintered or precipitated catalysts, however, showed significantly higher usage ratios in the unpromoted state. The presence of kieselguhr in precipitated

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90/ Weintraub, M., and Leva, M., Pressure Drop in Packed Tubes - A Nomogram: Chem. Eng. Prog., vol. 44, 1948, pp. 801-802.
91/ Leva, M., Fluid Flow Through Packed Beds: Chem. Eng., vol. 55, 1949, p. 115.
92/ Leva, M., Grummer, M., Weintraub, M., and Pollchik, M., Introduction to Fluidization: Chem. Eng. Prog., vol. 44, 1948, pp. 511-520.
93/ Leva, M., Grummer, M., Weintraub, M., and Pollchik, M., Fluidization of Nonvesicular Solid Particles: Chem. Eng. Prog., vol. 44, 1948, pp. 619-626.
94/ Leva, M., Weintraub, M., Grummer, M., and Pollchik, M., Fluidization of an Anthracite Coal: Ind. Eng. Chem., vol. 41, 1949, pp. 1206-1212.

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catalysts leads to high-usage ratios, probably as a result of adsorption of the promoter by the catalyst support. An investigation is being made to determine the effect of synthesis-gas ratio on the usage ratio obtained with fused synthetic-ammonia type catalysts.

In addition to the oil-circulation process, a study is being made of the oil-catalyst slurry process. In the latter process, a powdered catalyst is suspended in an oil that is nonvolatile under operating conditions, and the synthesis gas is bubbled up through this suspension. In this process, the boiling range of the liquid product can be varied over wide limits by selection of catalyst and operating conditions. The oil-catalyst slurry process has the additional advantage of possible continuous removal of partly spent catalyst and continuous introduction of regenerated or fresh catalyst. Laboratory and pilot-plant work on the oil-catalyst slurry process has included a large number of tests to determine the effects of rate of gas flow and of mechanical agitation on the distribution of the catalyst powder. Earlier experiments had shown the necessity of uniform distribution of the catalyst; even a small percentage of settling in any part of the reactor results in a marked decrease in activity and durability. A study of the utility of promoted naturally occurring iron ores (for example, goethite, FeO(OH)) for the slurry process was also begun; a method has been developed for separating goethite from acid-soluble ferric oxide.95/

To measure accurately the effect of the area of the gas to liquid interface, a study has been started in which a finely divided spray or fog of oil-catalyst slurry is injected into the synthesis gas. In addition to determining the importance of the gas-to-liquid interfacial area, these tests should show whether a spray or fogging type of operation is preferable to the bubbling of synthesis gas up through a column of oil-catalyst slurry.

The reduction of fused-iron catalyst is a relatively expensive step, because almost pure hydrogen is required, and the time and temperature (about 450° C.) of reduction necessitate a special installation for this operation. Precipitated catalysts can be inducted into the synthesis by treatment with synthesis gas at synthesis temperatures (about 250° C.), but this procedure has so far been found to be inapplicable to fused catalysts. Prolonged laboratory durability and activity tests are in progress to determine the effect of various degrees of reduction of iron catalysts and of impurities such as carbon oxides, methane, and water vapor in the hydrogen gas used for reduction. The results indicate that a high degree of reduction is essential for the fused iron oxide catalysts; almost complete reduction is obtained at 500° to 550° C., but the effect of pressure on the extent of reduction is less pronounced.

Carbon dioxide was shown to be about twice as effective as water vapor in decreasing the rate of reduction, whereas water vapor is more effective than carbon monoxide in this respect. At least part of the carbon monoxide and carbon dioxide is hydrogenated to methane. Methane had no effect upon reduction rate. These tests are of interest to the Demonstration Plant at Louisiana, Mo., for establishing conditions of catalyst reduction.

The unreduced fused-iron catalyst has only a very small surface area and little or no pore volume. Reduction at 450° C. provides a system of pores and fissures and

95/ Hawk, C. O., Golumbic, N., and Hofer, L. J. E., Catalyst for Hydrocarbon Synthesis: U. S. Patent 2,449,071, Sept. 14, 1948.

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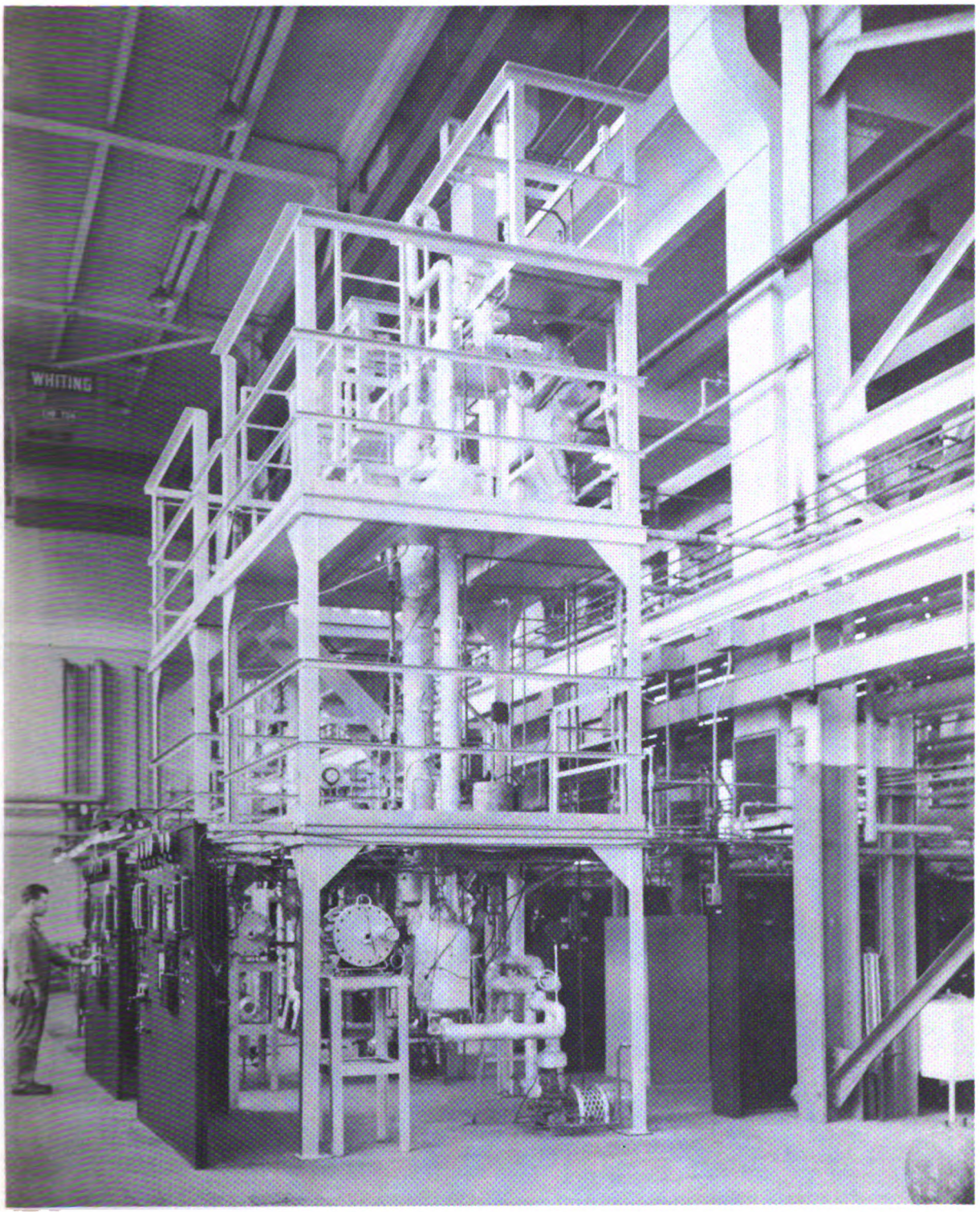

Figure 37. - Pilot plants for internally cooled (by oil circulation) process for synthesis of liquid fuels from hydrogen and carbon monoxide.

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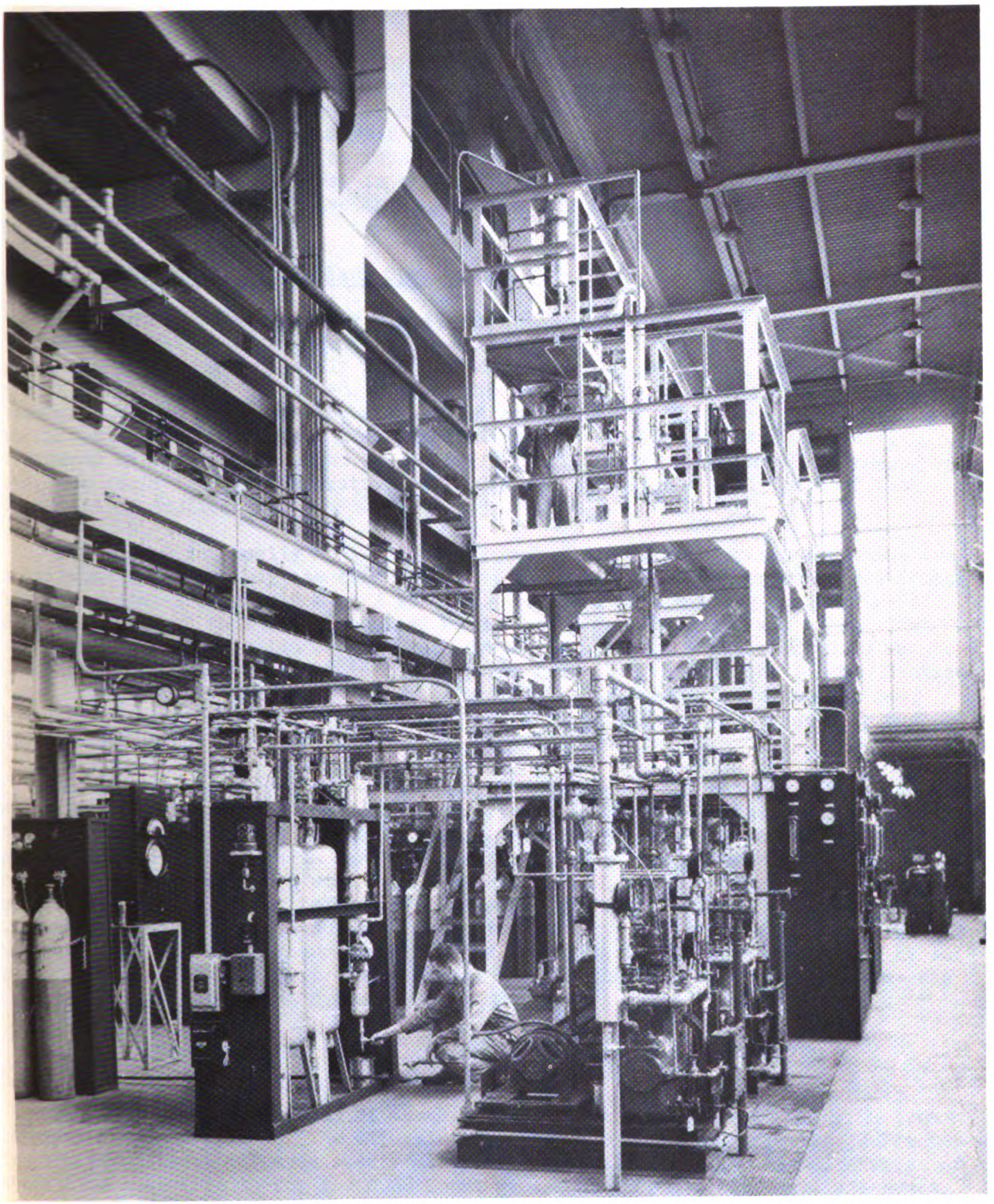

Figure 38. - Pilot plants for internally cooled (by oil circulation) process for synthesis of liquid fuels from hydrogen and carbon monoxide.

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an increased surface area. The unreduced precipitated catalysts have a relatively large area and pore volume and do not need extensive reduction before use in the synthesis. The sintered catalysts prepared from iron ores or mill scale resemble the fused catalysts, so far as reduction is concerned. However, the induction of a promoted goethite ore catalyst in synthesis gas at 325° C. has been found to result in a catalyst nearly as active as one reduced in hydrogen. Laboratory experiments are continuing this work.

Process Development, Pilot-plant Operations

A duPont synthetic-ammonia fused-iron catalyst was operated for about 3 months in the Fischer-Tropsch synthesis in the internally cooled (by oil circulation) process that uses a completely flooded bed of granular catalyst. The pilot plants are shown in figures 37 and 38. Operation was finally discontinued because of excessively high pressure drop across the catalyst bed. The operating temperature at this time was 269° C. for a carbon dioxide-free contraction of 59 percent. Treatment of the catalyst with hydrogen did not reduce the pressure drop through the bed, nor could the low temperature (255° C.) achieved in the early part of the synthesis be duplicated.

The second internally cooled converter was placed in operation, and these two units were used to study the effect of catalyst grain size and synthesis gas composition. Fused-iron catalyst of 2 to 4 mesh required an operating temperature of 272° C., compared to 255° C. required when 4- to 6-mesh catalyst was used. The addition of carbon dioxide to the fresh synthesis gas, the purpose of which was to simulate an unscrubbed synthesis gas prepared from coal, showed that carbon dioxide had no effect other than as a diluent. The use of hydrogen-rich synthesis gas, however, decreased not only the formation of acid in the converter but also the production of the heavier oils in favor of lighter hydrocarbons.

Because of the difficulties encountered in plugging the catalyst in the internally cooled converter, it was decided to try a modification of this design in which the catalyst is lifted by increased liquid velocity and kept suspended in a stream of liquid and gas. This design has been designated as a "moving-bed" converter. The result of preliminary work with this unit has shown that, as compared to fixed-bed operation, the moving-bed technique has the following advantages: (1) Greater space-time-yield, based on catalyst bed or converter height; (2) greater product yield on the basis of pounds of product per pound of catalyst; (3) elimination of the cementing of catalysts and large pressure drops; and (4) the possibility of treating the catalyst continuously with hydrogen in a side stream, should this be desired.

Separation and Identification of Products of Hydrogenation of Carbon Monoxide

Detailed characterization of the Fischer-Tropsch products obtained from iron catalysts is in progress. The determination of the oxygenated compounds in Fischer-Tropsch products by infrared spectrometry has been facilitated by the derivation of absorption coefficients for the functional groups of alcohols, ketones, aldehydes, esters, and acids in mixtures with hydrocarbons. This has minimized the previous requirements that pure calibration compounds be available. Thus, the limitations due to differences in molecular weight and density have been reduced, and calibrations based on the simpler C_4 oxygenated compounds can now be used to analyze oxygenated

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compounds in the C_{10} and higher ranges. Similar calibration work is in progress in regard to the functional group analysis of olefins.

Information concerning the molecular-weight distribution in the wax and heavy oil product from the Fischer-Tropsch process can be obtained by distillation at reduced pressures if reliable data concerning the vapor pressure of organic compounds of high molecular weights are available. Such information has heretofore been lacking for compounds above about C_{20} in the paraffin series. To supply this information, an alignment chart was prepared, which, if the vapor pressure of a paraffin at a particular temperature is known, permits an estimation of its molecular weight and vapor pressure at any other temperature. In connection with the evaluation of distillation columns operating at reduced pressures,96/ the use of a hydrocarbon whose hydrogen atoms are of mass one and the same hydrocarbon containing some deuterium atoms (hydrogen atoms of mass two) is highly desirable. Accordingly, a method of analysis for deuterated hydrocarbon was developed97/ that is superior to the analytical methods previously used.

SYNTHETIC LIQUID FUELS DEMONSTRATION PLANTS

The coal-to-oil demonstration plants were formally dedicated May 8, 1949. During the first half of May, more than 3,600 persons, representing industry, Government, and local residents, visited the plants and were taken on conducted tours during which the salient features were pointed out. Prior to dedication, both plants were operated to a limited extent. Lignite tar was hydrogenated to produce Diesel fuel which powered a train from St. Louis to Louisiana, Mo., and return. Gasoline produced at the same time is being used in all plant automobiles. The gasifier in the gas-synthesis plant was operated to produce an acceptable synthesis gas. Nevertheless, the construction program has not yet been completed, and details of status are shown in figures 39 and 40.

Coal Hydrogenation Plant

The process flow diagram of the 200- to 300-barrel-per-day capacity plant has been published.98/ More detailed discussion of the process flow, equipment, construction, and operation of the plant appeared in a recent publication.99/ Engineering design was completed in December 1948, and construction work by the Bechtel Corp. ended in January 1949. The testing of the units and finishing of odds and ends was undertaken by the station's maintenance and operating forces.

96/ Feldman, J., Myles, M., Wender, I., and Orchin, M., Evaluation of Vacuum Rectification Columns. Use of Binary Mixtures: Ind. Eng. Chem., vol. 41, 1949, pp. 1032-1036.

97/ Wender, I., Friedel, R. A., and Orchin, M., Preparation of High-Purity Hydrogen Deuteride from Lithium-Aluminum Hydride: Jour. Am. Chem. Soc., vol. 71, 1949, p. 1140.

98/ Fieldner, A. C., and Gottlieb, Sidney, Annual Report of Research and Technologic Work on Coal, Fiscal Year 1948: Bureau of Mines Inf. Circ. 7518, 1949, 87 pp.

99/ Kastens, Merritt L., assoc. ed., (in collaboration with L. L. Hirst and C. C. Chaffee, Bureau of Mines, Louisiana, Mo.), Liquid Fuel from Coal: Ind. Eng. Chem., vol. 41, 1949, pp. 870-885.

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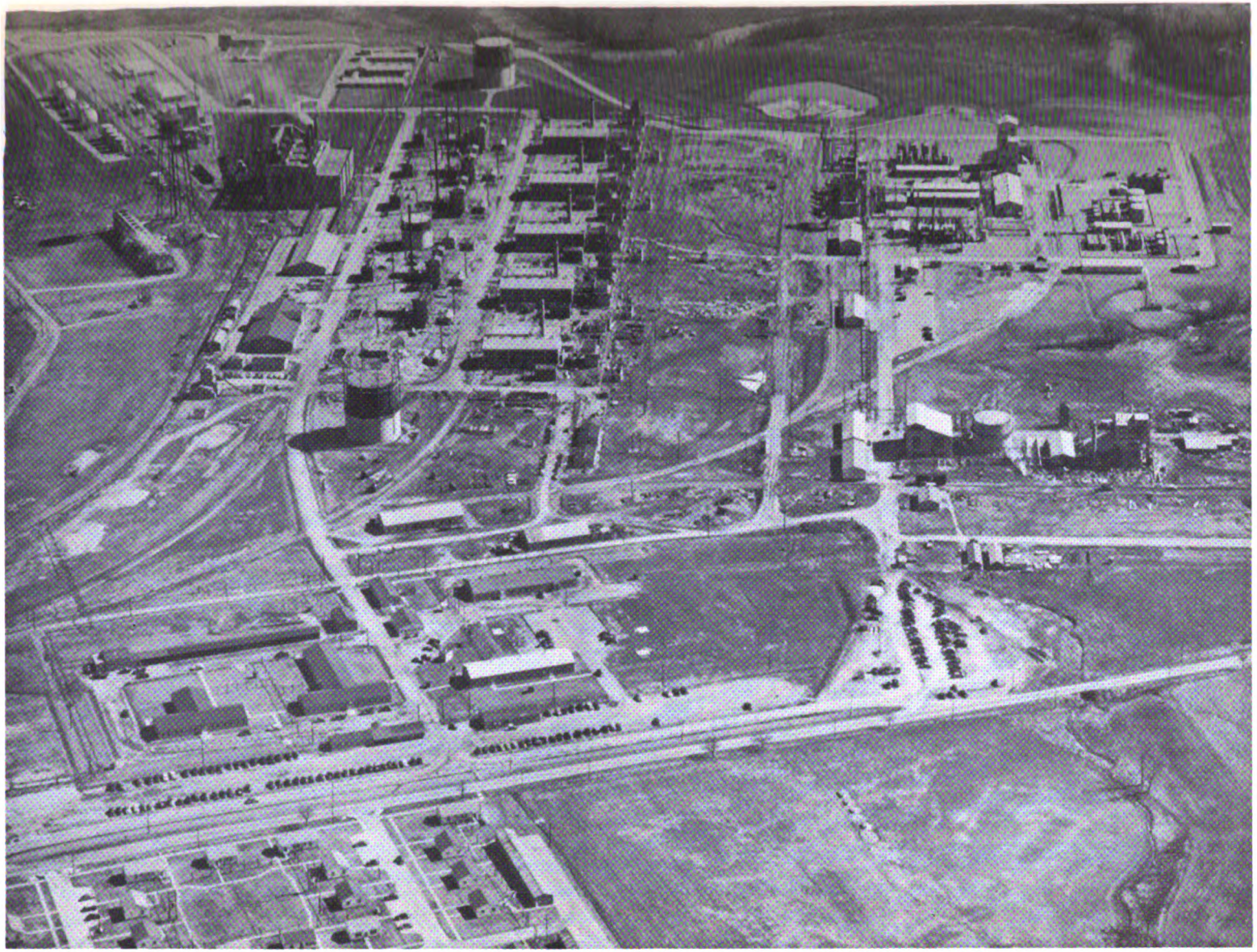

Figure 39. - Air view of coal-to-oil demonstration plants at Louisiana, Mo. Missouri Ordnance Works, upper left; hydrogenation plant, upper right; gas-synthesis plant, lower right; administration buildings and employee housing development, lower left.

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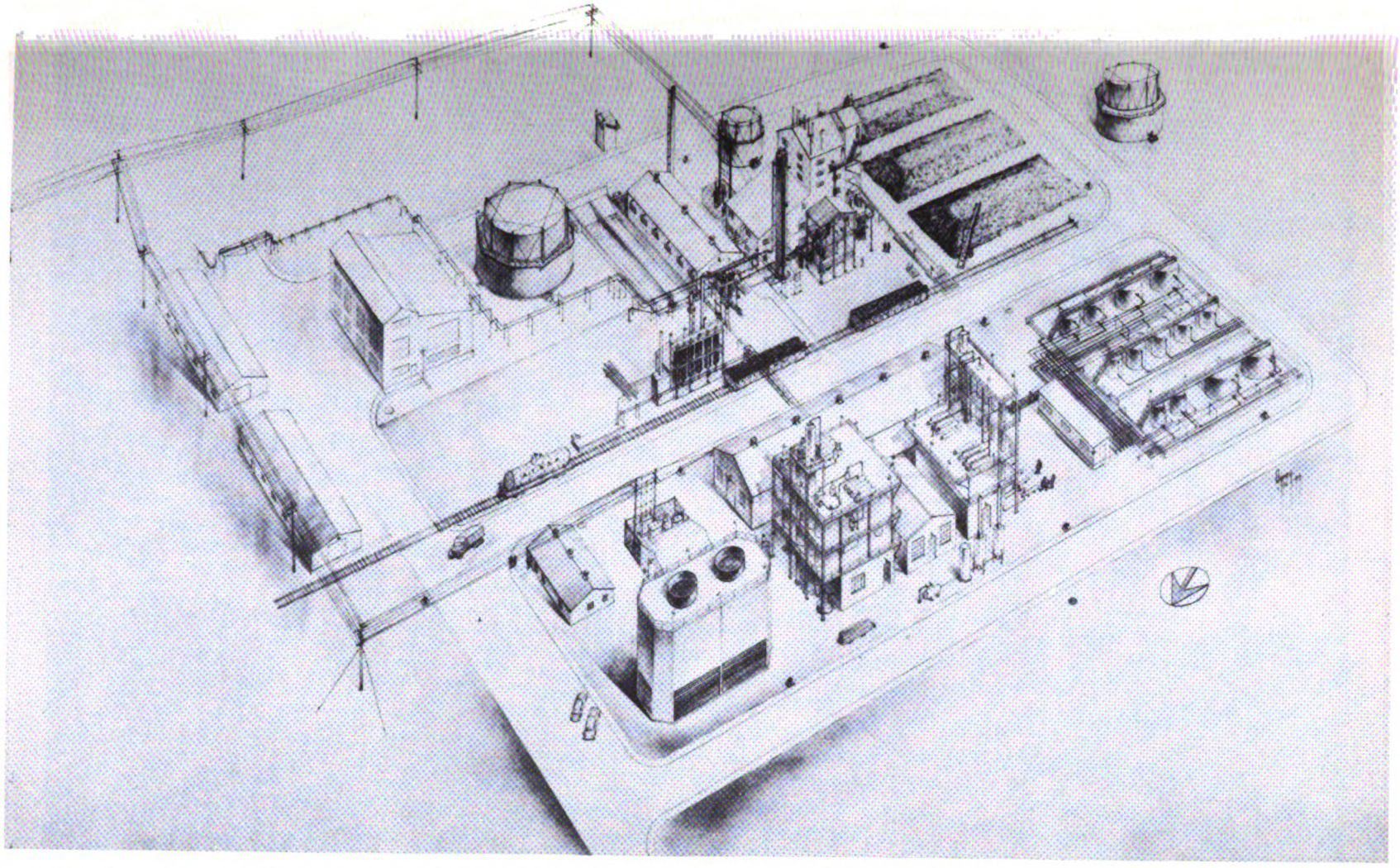

Figure 40. - Perspective sketch of the Bureau of Mines gas synthesis demonstration plant under construction at Louisiana, Mo. As this plant employs a modified Fischer-Tropsch process to convert coal to oil, this plant will have a capacity of 80 to 100 barrels daily. Left to right above the railroad tracks are the oxygen and coal gasification units; below the tracks are the synthesis units and the product storage area.

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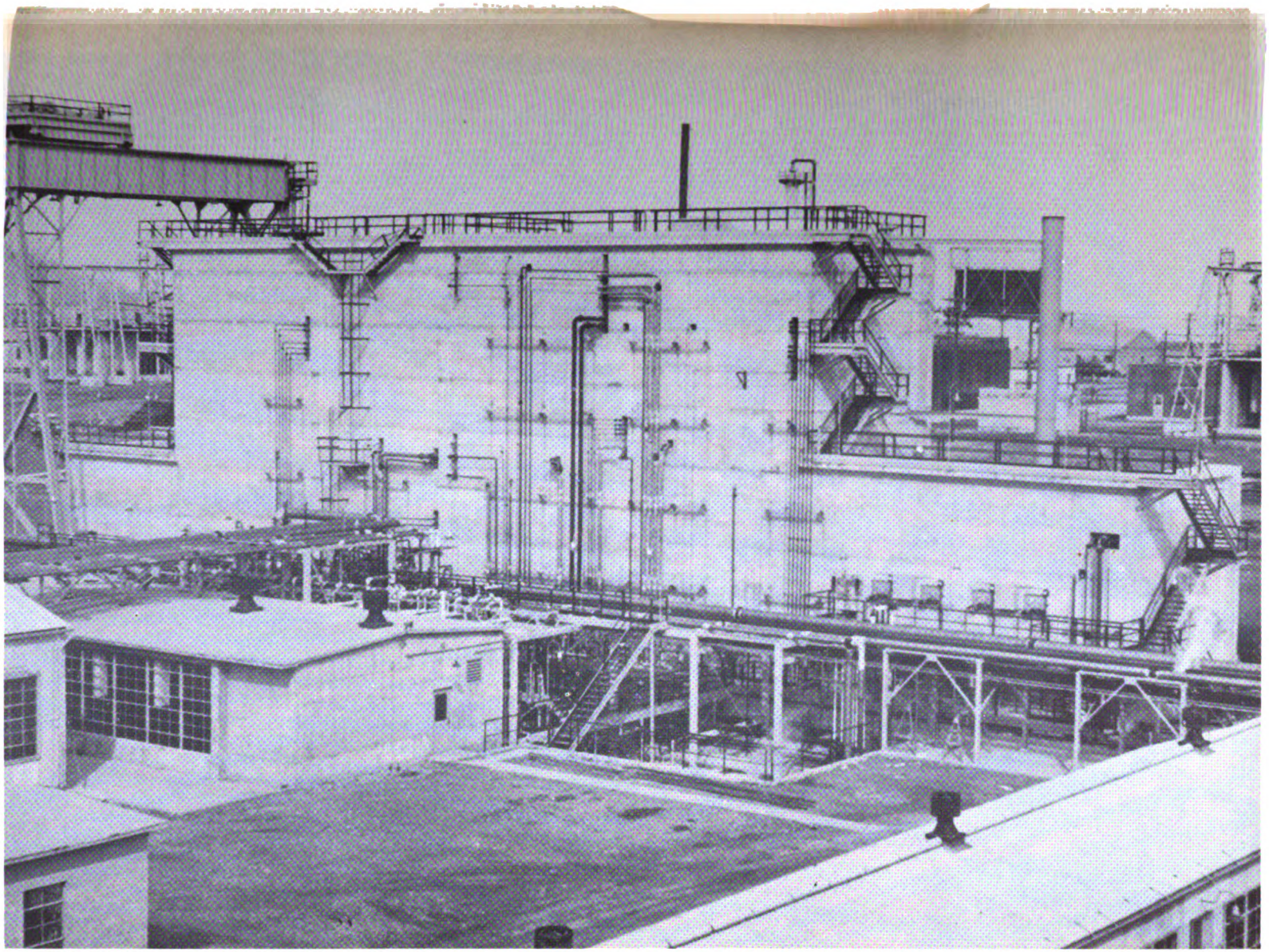

Figure 41. - This large reinforced-concrete structure is partitioned, for the protection of operators and equipment, into stalls enclosing on three sides the high-pressure and temperature vessels in which coal is converted to oil. The 130-ton traveling Gantry crane at left straddles the stalls and can carry the largest vessel. Squat building nearest the stalls is the control house.

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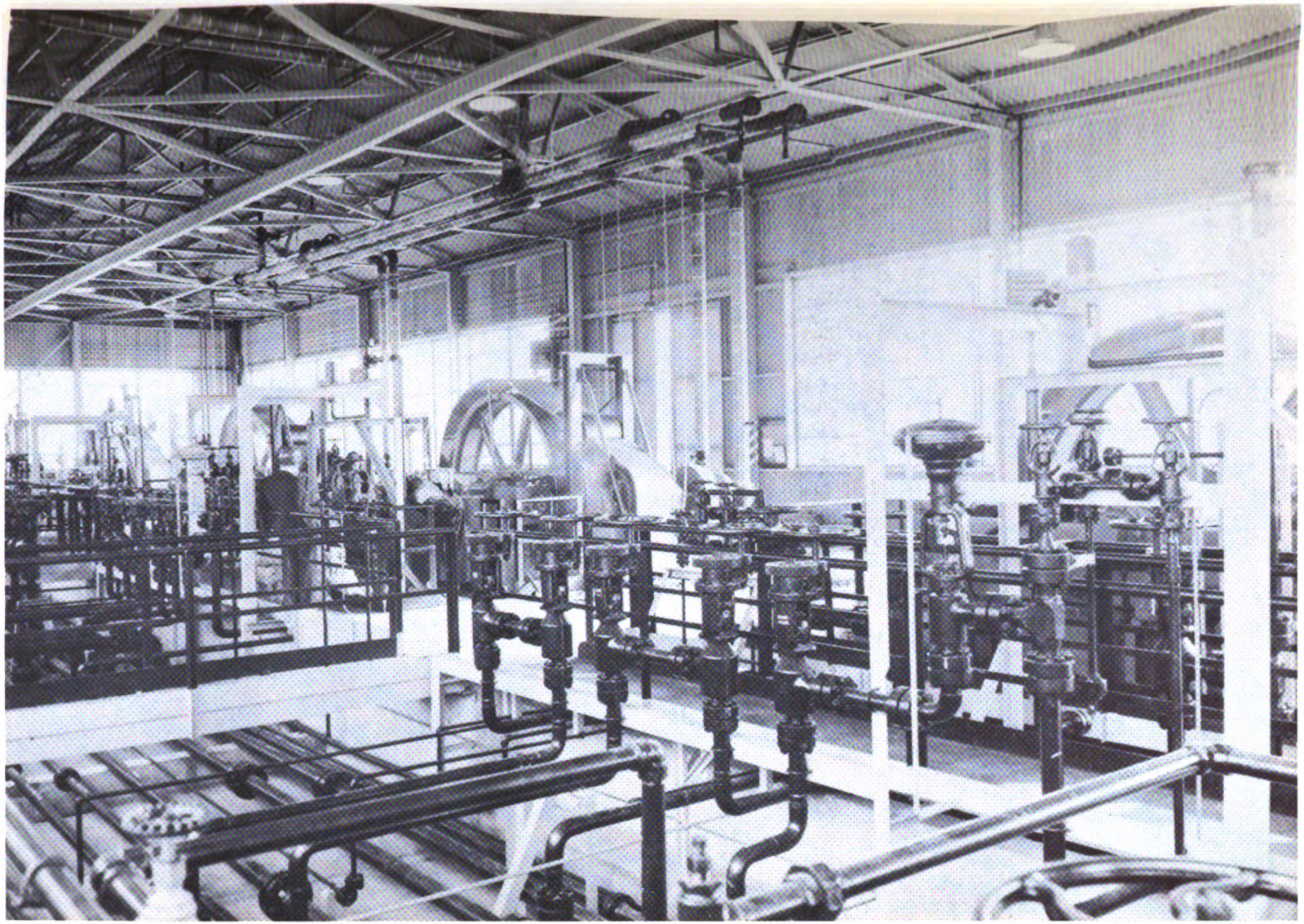

Figure 42. - In the compressor house, four high-pressure compressors return unused hydrogen to the conversion vessels, where it serves both as a cooling agent and as an additive to the coal. Two of the compressors are driven by electric motors and two by steam turbines.

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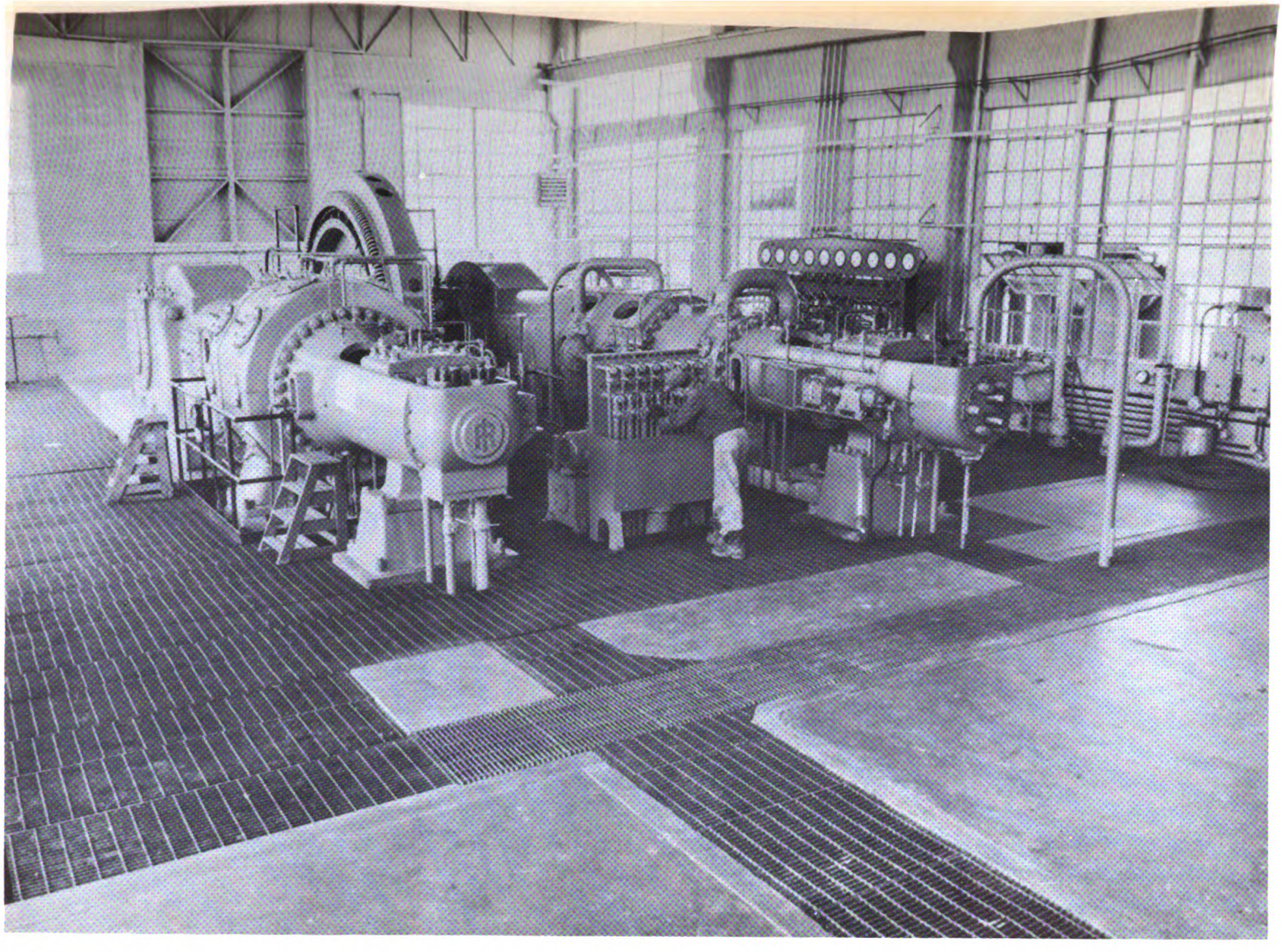

Figure 43. - One of the huge hyper compressors in the Missouri Ordnance Works now employed to replenish the hydrogen used in converting coal to oil. A seven-stage machine, it can compress 210,000 cubic feet of hydrogen per hour from atmospheric pressure to 15,000 pounds per square inch.

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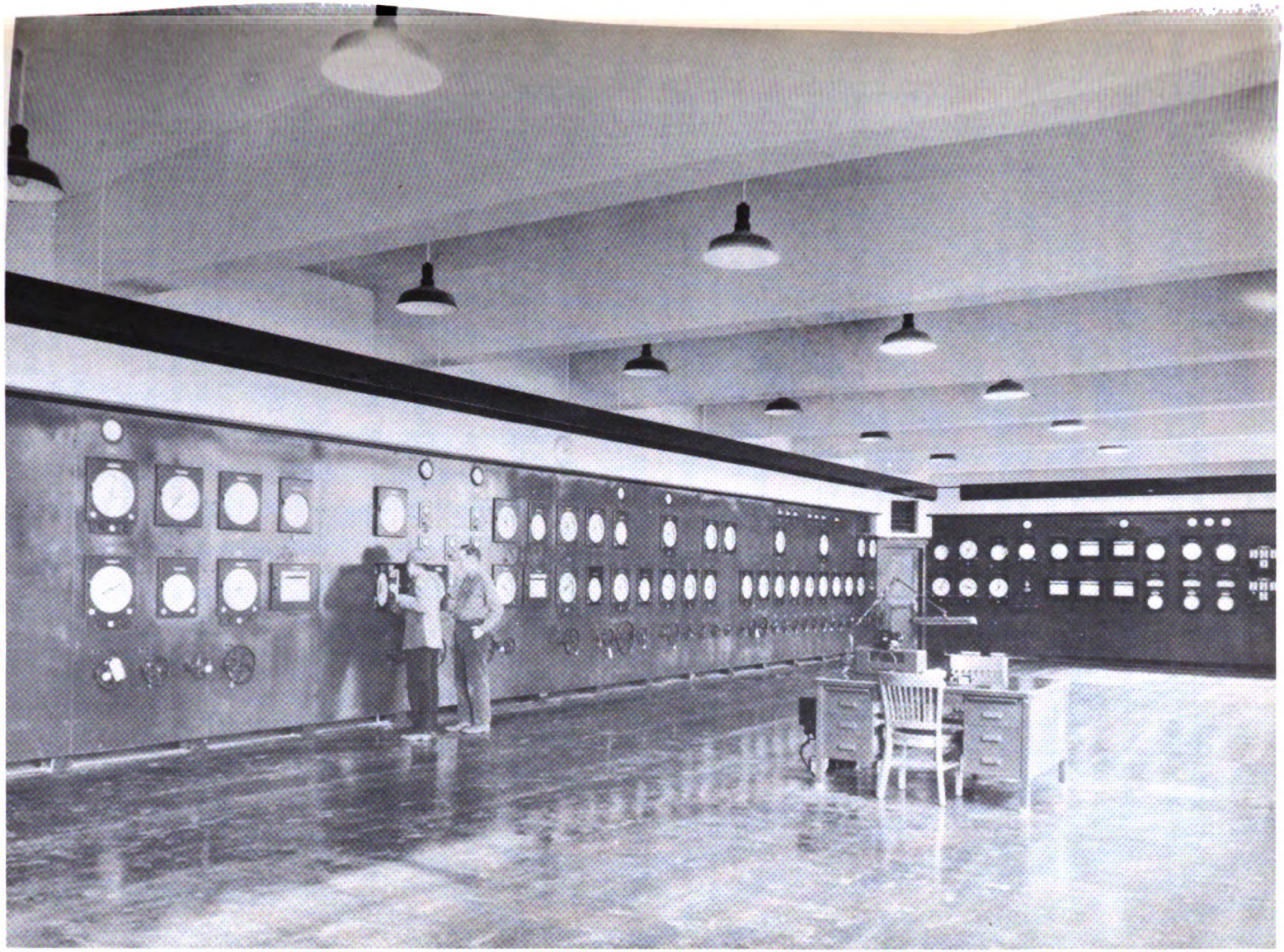

Figure 44. - The control house in the Coal Hydrogenation Demonstration Plant. Here, unerring instruments keep watch day and night over complex operations, observing and recording all pertinent changes in temperatures, pressures, and flows. No high-pressure lines enter this building, for the instruments transmit their messages by variable electric current or by air and liquid streams.

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Although actual hydrogenation of coals has not yet been attempted, successful unit operations leading toward full plant operation have been carried out.

The gas-manufacturing area (part of the former Missouri Ordnance Works) was reactivated for the purpose of testing the equipment for hydrogen and nitrogen manufacture and for training the newly recruited, inexperienced operating personnel. This part of the plant also was operated intermittently, as required, during February and March for the purpose of supplying high-pressure nitrogen required in testing the high-pressure equipment in the hydrogenation plant. In April, hydrogen was manufactured, purified, and compressed as required for the first vapor-phase hydrogenation run.

The distillation area or refinery was taken over from the contractor late in 1948 for break-in operation on petroleum. Two months' operation required for testing the equipment, further training of operators, and preparation of petroleum-oil fractions for later hydrogenation break-in runs were completed successfully in February 1949.

The Bureau's operators and maintenance forces took over the high-pressure testing work of the hydrogenation plant in January 1949 (figs. 40, 41, 42, 43, and 44). High-pressure testing was carried out in February and March simultaneously with completion of wiring and zeroing instruments, process and steam piping, electrical installations, line bracing, insulation and painting. All testing was completed in the vapor phase unit and a run was made in April which successfully accomplished the following objectives:

1. It was determined that the unit as designed was operable.

2. The operating training program was found to be adequate.

3. Light petroleum oil was hydrogenated successfully at 700 atmospheres to produce motor gasoline.

4. Coal-tar distillate from North Dakota lignite was hydrogenated successfully at 700 atmospheres to produce high-octane gasoline and specification Diesel fuel.

This operation is of particular interest because it represents the successful run of the first 700 atmospheres hydrogenation plant in this country. It is also noteworthy that the run was made without delay or serious interruption, although the plant had not been run as a whole previously, and none of the operating crew had actual experience with similar operations.

The liquid-phase hydro plant has withstood high-pressure tests satisfactorily, and operation will be started when repairs and changes to injection pumps, nozzle welded piping, and instruments are completed.

Plant-scale coal preparation suitable for hydrogenation purposes is a new problem in this country. The coal must be dried to a low moisture content at low temperatures (300°-400° F.) in an inert-gas stream having low oxygen content to

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prevent oxidation and distilling of tar from the coal. Changes to equipment also were necessary, so that the dried coal would contain a minimum of coarse and extremely fine material and yet meet all other requirements as to quality.

These process limitations have required considerable testing and changing of the coal-preparation unit during the spring and summer of 1949. The unit is now ready for operation and is capable of preparing 5 to 8 tons of specification pulverized coal per hour.

During the early spring of 1949, approximately 70,000 gallons of coal tar pasting oil and 10,000 gallons of solid-free, light, aromatic, flushing oil were obtained for the purpose of breaking-in the heavy-oil storage and pasting equipment and for initiating the liquid-phase coal operations. Intensive operator training and break-in runs of the flash distillation-area equipment, heavy oil-blending equipment, paste making, and centrifuges are now in progress.

Gas-Synthesis Plant

For the gas-synthesis process, coal must first be converted to a hydrogen-carbon monoxide gas mixture and this, in turn, converted to liquid products. Transforming coal into gas can be approached in several ways. At Louisiana, Mo., a method is being tried that reacts powdered coal with steam and oxygen. Two processes are contemplated for making finished synthetic fuel products from the synthesis gas.

As shown in the block diagram (fig. 45), the plant is composed of five parts, namely, oxygen production, gasifier, purification, Fischer-Tropsch-synthesis, and refinery.

The oxygen plant, a Linde-Frankl unit brought from Germany, produces 1 ton per hour of oxygen of 98 percent purity.

The coal-handling facilities are intended to dry run-of-mine coal and reduce it to approximately 200-mesh, after which it is conveyed pneumatically and stored in bulk hoppers, wherefrom it is fed to the gasifier system.

The gasifier uses coal, oxygen, and steam superheated to 2,500° F. It will make up to 90,000 cubic feet an hour of synthesis gas. The reaction vessel is a refractory-lined steel shell 6-1/2 feet in inside diameter by 9 feet inside length. Design rates are 2,300 pounds of coal, 2,000 pounds of oxygen, and 2,900 pounds of superheated steam per hour.

After leaving the gasifier, the gas is cooled in a waste-heat boiler, water-scrubbed, and then put through an electrical precipitator for the removal of dust. Sulfur compounds in the gas are removed by a series of treatments at pressures of 300 to 450 pounds per square inch. The allowable dust and sulfur compounds are in the magnitude of tenths of grains for 100 cubic feet of gas.

In the synthesis section of the plants, this gas is treated catalytically to form it into synthetic-fuel raw products. In the demonstrated process, the cooling oil will carry away the generated heat from the fixed catalyst bed of the reactor. Following this, the liquid products will be refined into specification-grade gasoline, Diesel oil, and waxes. Facilities will be available for distilling the gross liquid product into its component commercial fractions and for converting the heavy gases to

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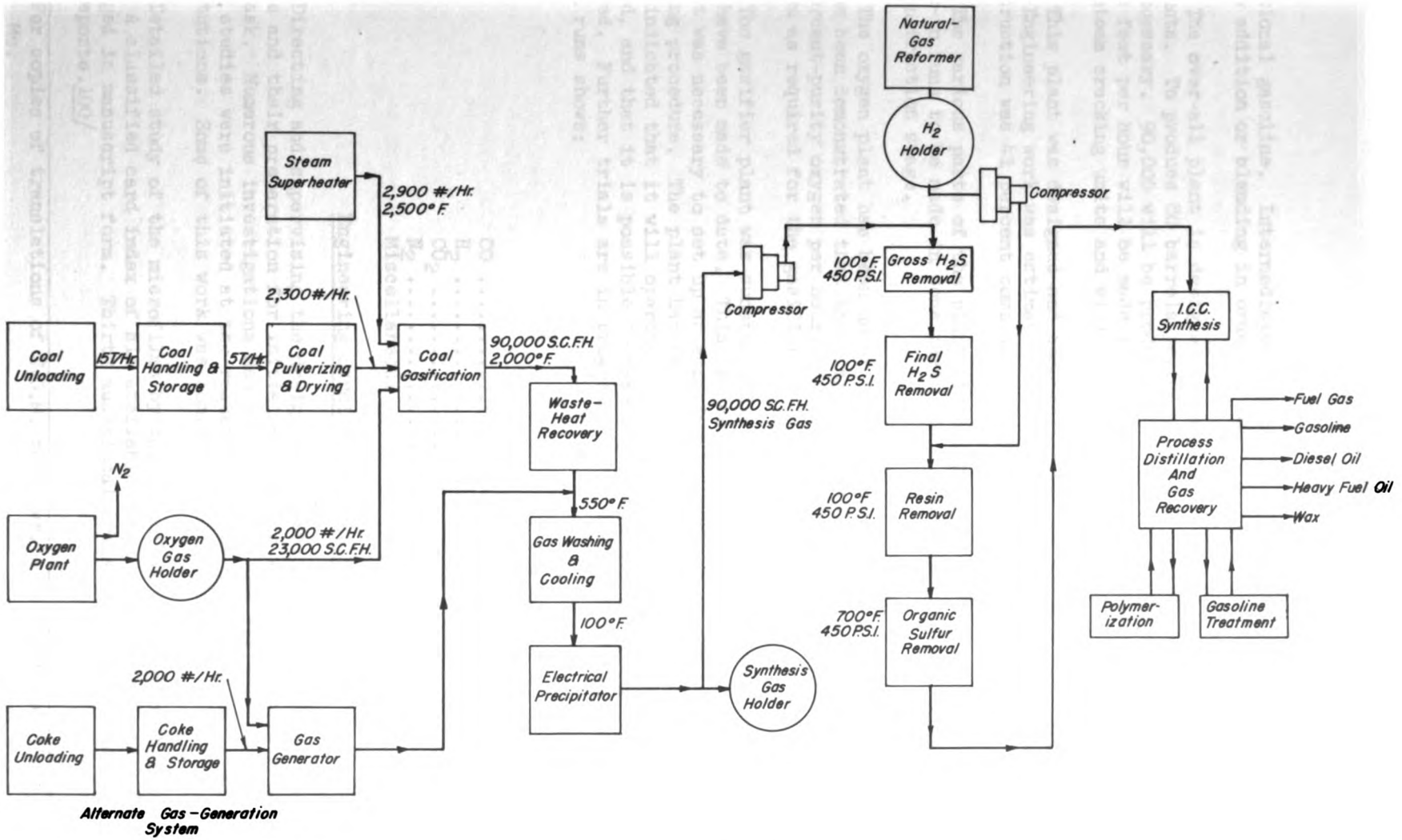

Figure 45. - Block diagram of flow, Gas Synthesis Demonstration Plant.

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additional gasoline. Intermediate products will be upgraded by catalytic treatment or by addition or blending in order to make high-quality final products.

The over-all plant is designed to make 50 to 80 barrels a day of total finished products. To produce 80 barrels per day, 140,000 cubic feet per hour of synthesis gas is necessary. 90,000 will be produced in the Koppers gasifier, the remaining 50,000 cubic feet per hour will be made in one of the former Missouri Ordnance Works natural gas-steam cracking units and will be blended with the products of the coal gasifier.

This plant was designed and constructed by the Koppers Co., Inc., of Pittsburgh, Pa. Engineering work was estimated to be 83 percent complete on June 15, 1949, and construction was 41 percent complete on May 31.

The various parts of the plant are being built successively, which permits break-in runs to be made in some parts while other sections are still in the design or construction stage.

The oxygen plant has been under intermittent operation since December 20, 1948. It has been demonstrated that the plant will produce its design capacity of 1 ton of 98-percent-purity oxygen per hour. At present it runs continuously and supplies oxygen as required for the gasifier.

The gasifier plant was substantially completed in May 1949, and several trial runs have been made to date. This process had never before operated on this scale, and it was necessary to set up an experimental program in addition to the normal testing procedure. The plant has been operated at full capacity, and early trials have indicated that it will operate according to the principle upon which it was designed, and that it is possible to make a gas that has the general composition desired. Further trials are in progress. A typical gas composition from one of the trial runs shows:

	Percent
CO	38
H_2	42
CO_2	16
N_2	3
Miscellaneous	1

Engineering Studies and Cost Estimates

Directing and supervising the design and construction of the two demonstration plants and their preparation for initial operations was a continuous major engineering task. Numerous investigations had to be carried out for this purpose, and many other studies were initiated at the request of other Government agencies and technical institutions. Some of this work was completed and published.

Detailed study of the microfilms by the Technical Oil Mission to Europe continued. A classified card index of all available reels was prepared and soon will be arranged in manuscript form. Thirty additional translations were made and issued as "T" reports.100/

100/ For copies of translations of T.O.M. reels write to Bureau of Mines, Louisiana, Mo.

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Information regarding commercial-size synthetic-fuel plants was supplied to the Department of the Army, Corps of Engineers, to assist them in their survey to find suitable commercial plant site areas.

In a paper,101/ the thermal efficiency of an ideal coal-hydrogenation plant was discussed in detail, and the efficiency obtainable was compared with that of a typical German hydrogenation plant. A number of new ideas and processes are discussed and incorporated into the process flow to improve the over-all heat efficiency from 28.9 percent for a typical German plant to a figure of approximately 55 percent.

Later, coal-hydrogenation improvements incorporated in a preliminary plant design102/ indicated a calculated thermal efficiency of 48 percent. Major proposed improvements over the German processes include the gasification of coal under pressure for hydrogen production and delayed coking of residue for greater oil recovery.

A report of investigations on the subject of estimated cost of producing heavy fuel oil by hydrogenation of coal was prepared.103/ The report contains process information and cost figures that indicate that fuel oil can be produced from coal at a cost of approximately $4 per barrel in a 10,000-barrel-per-day plant using a coal costing $3 per ton at the mine.

Economic studies are always of extreme importance when the feasibility of a new process or operation is considered. A report has been written104/ to help those who desire to use a method of making uniform cost estimates for the following purposes:

1. To make an economic study of the operations of plants and equipment of new design.
2. To make economic studies of new products and methods of producing them.
3. To include all elements of operating cost, such as direct, indirect, fixed costs, etc.
4. To present the various cost elements so arranged that they may be compared with other cost estimates of like or similar nature.

101/ Skinner, L. C., Dressler, R. G., Chaffee, C. C., Miller, S. G., and Hirst, L. L., Thermal Efficiency of Coal Hydrogenation: Ind. Eng. Chem., vol. 41, 1949, p. 87-95.

102/ Hirst, L. L., Skinner, L. C., and Donath, E. E.: Improvements in Hydrogenation of Coal: Bureau of Mines Inf. Circ. 7486, 1948, 7 pp.

103/ Hirst, L. L., Skinner, L. C., Clarke, E. A., Dougherty, R. W., and Levene, H. D., Estimated Cost of Producing Heavy Fuel Oil by Hydrogenation of Coal: Bureau of Mines Rept. of Investigations 4413, 1948, 53 pp.

104/ Van Noy, C. W., Dunville, T. C., Dressler, R. G., and Chaffee, C. C.: Guide for Making Cost Estimates for Chemical-Type Operations: Bureau of Mines Rept. of Investigations 4534, 1949, 64 pp.

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The Bureau of Mines' synthetic-fuels program was discussed in a general way, and the demonstration plants were described in some detail to the American Gas Association.105/

Technical Reports and Foreign Document Work

Synthetic Liquid Fuels Abstracts

The current literature has been covered and abstracted thoroughly; German patent references in the foreign-document files have been searched, and those of interest have been abstracted. Over a thousand abstracts have been made and published, and about 1,000 copies of each issue of the abstracts have been distributed. Indexes of all abstracts issued since October 1944 have been made, thus opening for easy inspection all of the synthetic-fuels literature issued since that date.

Bibliographies

A review and compilation of the literature on the pressure hydrogenation of liquid and solid carbonaceous materials is in progress.

The Fischer-Tropsch bibliography is still in the formative stage, the card index is being completed, and the closing date for the manuscript will be moved ahead as far as possible so that the current literature references and the German patent abstracts from the foreign documents can be added. It is anticipated that this bibliography will contain about 5,000 abstracts in its completed form.

Foreign Documents

Work has been continued throughout the past year in collecting, arranging, classifying, and indexing the foreign documents from German sources containing information of interest on synthetic liquid fuels. Individual subject indexes of the 279 TOM reels have been prepared, and these are now being consolidated into a single index. Enlargement prints selected from the TOM reels and printed CIOS, BIOS, and FIAT reports, as well as translations of documents from various sources, are available. Documents other than TOM reels are being indexed in considerable detail, and an attempt will be made to correlate this index with the TOM reel index. The document index now covers more than 1,500 separate documents of specific interest to synthetic liquid fuels and composes 10 standard file drawers of index cards. Seven complete sets of these cards have been prepared by means of ditto reproduction and placed in advantageous places for reference. It is anticipated that when this index is completed, reference will be made to every document or report from German sources that contain specific information of interest in connection with synthetic liquid fuels.

105/ Hirst, L. L., Markovits, J. A., Dressler, R. G., and Batchelder, H. R., The Bureau of Mines Demonstration Plants in the Synthetic Fuels Picture: Proc. Am. Gas Assoc., 1948, pp. 317-325.

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